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THE INFLUENCE OF MILK FAT DISPERSION IN CREAM ON THE THIXOTROPIC PROPERTIES OF CONVENTIONAL BUTTER

by

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The undersigned certify that they have read, and recommend to the Faculty of Graduate Studies for acceptance, a thesis entitled

THE INFLUENCE OF MILK FAT DISPERSION IN CREAM
ON THE THIXOTROPIC PROPERTIES OF CONVENTIONAL BUTTER

submitted by Phyllis Bakar in partial fulfilment of the requirements for the degree of Master of Science.

ABSTRACT

The decreased size of the milk fat globules in homogenized cream appeared to be an important factor influencing the accelerated setting rate of butters held at 55.5°F after manufacture, although the final butter hardness, measured after 30 days' storage at 40°F, was not notably increased by homogenizing of cream. This finding is similar to that reported for butter made from Vacreator-pasteurized cream.

The increase in setting of butters churned from homogenized cream was accentuated by holding of the cream for only one hour at 40°F before churning. Both continuing crystallization and polymorphic transformations may contribute to the exceptionally rapid setting of these butters during the first few hours after manufacture. However, it is likely that the major factor involved in the increase in setting of butters from both Vacreator-pasteurized and homogenized creams is the larger amount of thixotropic crystals in the butters.

Slow cooling, precooling and stepwise cooling of homogenized cream seemed to have unusual effects on butter setting and hardness compared to those found for Vacreator-treated cream. It appears that these discrepancies are caused, at least in part, by differences in the mode of milk fat crystallization in Vacreator-pasteurized and homogenized creams.

Addition of increasing amounts of butteroil to cream before cooling proved ineffective in changing either the setting rate or the final hardness values of butters from homogenized or unhomogenized creams;

the added fat appeared to coalesce into aggregates. These results may indicate that the presence of a slightly larger number of coalesced milk fat globules in Vacreator-pasteurized cream is of little consequence as a factor affecting the physical properties of butter.

Penetration, sectility and extrusion procedures were used to determine the hardness of sixty samples of conventionally-churned butter and twenty samples of Gold'n Flow continuously-made butter. The extrusion method compared favorably with the penetration and sectility procedures for measuring butter hardness.

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TABLE OF CONTENTS

		Page
I	INTRODUCTION	1
II	REVIEW OF LITERATURE	3
	1. Structure of conventional butter	3
	2. Crystallization of milk fat	5
	3. Hardness of butter and setting phenomenon	9
	4. Mechanism of setting	10
	5. Factors affecting hardness and setting of butter	12
	6. The thixotropic phenomenon	14
	7. Measurement of the hardness of butter	18
III	EXPERIMENTAL METHODS	23
	1. Cream processing	23
	2. Homogenizing of cream	24
	3. Cooling of cream	24
	a. Slow cooling	24
	b. Rapid cooling	25
	c. Precooling and stepwise cooling	26
	(i) Control treatment	26
	(ii) Precooled treatment	26
	(iii) Stepwise-cooled treatment	28
	4. Holding of cream	28
	5. Addition of liquid fat to cream	30
	6. Churning, working and sampling butter	30
	7. Determination of hardness and setting of butter	31
	8. Moisture content of butter	33
	9. Polarized light microscopy	35
IV	RESULTS	36
	1. Homogenizing of cream	36
	2. Rate of cooling cream	41
	3. Precooling and stepwise cooling of cream	45
	4. Addition of liquid fat to cream before cooling	45
	5. Time interval between cream cooling and churning	48
V	DISCUSSION	52
	1. Effect of homogenization of cream	52
	2. Influence of the cream cooling rate	54
	3. Effects of precooling and stepwise cooling of cream	55

TABLE OF CONTENTS (Continued)

		Page
V	DISCUSSION (Continued)	
	3. a. Precooling	55
	b. Stepwise cooling	55
	4. Addition of liquid butterfat to cream	57
	5. Influence of the time interval between cooling of cream and churning	58
VI	BIBLIOGRAPHY	60
VII	APPENDIX A	A-1
	1. Procedure for measuring butter hardness	A-1
	2. Relationship of three methods of measuring butter hardness	A-5
	3. Discussion	A-13

LIST OF TABLES

		Page
Table 1	Cooling rate data for 10 representative lots of cream	27
Table 2	Accuracy of hardness measurements with the modified Kruisheer and den Herder penetrometer . . .	32
Table 3	Increase in butter hardness attributable to homogenizing of cream	38
Table A-1	Accuracy of extrusion, penetration and sectility measurements	A-3
Table A-2	Correlation coefficients significant at 1% level for conventional and continuous butters tested for hardness by three methods	A-12

LIST OF FIGURES

		Page
Figure 1	Cream holding vats	29
Figure 2	NIRD extruder modified for penetration and sectility measurements	34
Figure 3	Effect of cream homogenizing temperature on setting and hardness of experimental butters	37
Figure 4	Setting and hardness of representative summer and winter butters from homogenized cream (H) and unhomogenized cream (U)	39
Figure 5A	Photomicrograph of butter from unhomogenized cream (500 x)	40
Figure 5B	Photomicrograph of butter from homogenized cream (500 x)	40
Figure 6	Setting and hardness of typical butters from rapidly cooled and slowly cooled unhomogenized cream	42
Figure 7A	Photomicrograph of butter from rapidly cooled unhomogenized cream (500 x)	43
Figure 7B	Photomicrograph of butter from slowly cooled unhomogenized cream (500 x)	43
Figure 8	Setting and hardness of typical butters from rapidly cooled and slowly cooled homogenized cream	44
Figure 9	Effects of precooling and of stepwise cooling of unhomogenized cream on setting and hardness of typical experimental butters	46
Figure 10	Effects of precooling and of stepwise cooling of homogenized cream on setting and hardness of typical experimental butters	47
Figure 11	Influence of time interval between cooling and churning of cream on setting and hardness of summer butters from homogenized cream (H) and unhomogenized cream (U)	50

LIST OF FIGURES (Continued)

		Page
Figure 12	Influence of time interval between cooling and churning of cream on setting and hardness of winter butters from homogenized cream (H) and unhomogenized cream (U)	51
Figure A-1	Typical traces of penetration, sectility and extrusion determinations	A-4
Figure A-2	Relationship between penetration and extrusion values for 60 samples of conventional butter . . .	A-6
Figure A-3	Relationship between penetration and extrusion values for 20 samples of continuous butter	A-7
Figure A-4	Relationship between extrusion and sectility values for 60 samples of conventional butter . . .	A-8
Figure A-5	Relationship between extrusion and sectility values for 20 samples of continuous butter	A-9
Figure A-6	Relationship between penetration and sectility values for 60 samples of conventional butter . . .	A-10
Figure A-7	Relationship between penetration and sectility values for 20 samples of continuous butter	A-11

I. INTRODUCTION

It has been shown by a number of investigators (Mulder, 1949; Huebner and Thomsen, 1957b; de Man and Wood, 1958a, 1958b) that the setting of conventionally churned butter (increase in hardness after the completion of working) is primarily a thixotropic phenomenon and is not the result of continuing crystallization. The rate of setting has been found to be rapid when the butter is maintained at 55°F, a normal temperature at the end of the working process (de Man and Wood, 1959c). Setting is virtually complete after storage of the butter at 40°F for one week, although it may continue at a slow rate for as long as two months.

Investigations by Dolby (1954, 1959) and Wood and Dolby (1965) have indicated that the method of cream pasteurization also influenced the rate of setting and the final hardness of butter. This effect of the pasteurizing method was first indicated by the work of Dolby, who reported that the precrystallization stage of the Alnarp cream cooling procedure was ineffective in reducing the hardness of New Zealand butter made from cream pasteurized in a Vacreator*. Subsequent studies by Wood and Dolby showed that butter churned from Vacreated cream set faster to higher hardness values than did butter from plate-pasteurized cream. This increased setting was most pronounced when a high steam intensity was used in the Vacreator treatment. Furthermore, this investigation confirmed the find-

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*Murray vacuum pasteurizer manufactured by Mauri Bros. and Thompson, Auckland, New Zealand.

ings of Samuelsson and Pettersson (1937) that the precrystallization step of the Alnarp cream cooling method was effective in reducing the final hardness of butter churned from non-Vacreated cream. The cause of these differences, which were attributed to the method of pasteurization, was not known. It was assumed, however, that the cream treatment in the Vacreator influenced the number of crystals of thixotropic dimensions present in the butter. An investigation by Dolby (1953) showed that Vacreated cream contained an increased number of milk fat globules less than two microns and greater than ten microns in diameter, and fewer globules of intermediate size. These observations indicate that the thixotropic setting of butter made from Vacreator-pasteurized cream may be influenced by this change in the size of the milk fat globules.

The present investigation was undertaken to determine the effect of processes influencing the fat globule size in cream on the setting and final hardness of butter. The appendix of the report contains the results of a limited study made to compare three methods of measuring butter hardness.

II. REVIEW OF LITERATURE

1. Structure of Conventional Butter

A number of early theories regarding butter structure were reviewed by King (1955). According to Van Dam and Burgers (1935), X-ray diffraction studies indicated that both solid and liquid fat were present in butter. The theory of King (1932) stated that conventionally-made butter contained fat globules, fat crystals, moisture droplets and air cells dispersed in a continuous phase of liquid fat. The liquid fat was thought to originate from fat globules of cream which were deformed or destroyed during the churning and working processes. King (1947) reported that normal butter contained 57-72% globular fat. Many of the fat globules in conventional butter were intact and appeared identical to the globules of cream.

Under polarized light, globules of milk fat in cream and butter appeared to be surrounded by a birefringent peripheral layer which was believed to consist of crystalline fat (King, 1932, 1962; de Man and Wood, 1959a). Walstra (1962) confirmed that the peripheral layer of fat globules in butter is birefringent due to optical anisotropy.

High melting glycerides have been regarded as normal constituents of the milk fat globule membrane. Recently, Vasic and de Man (1965a) presented evidence that the high melting glycerides in the peripheral layer were artefacts caused by crystallization of milk fat at the membrane surface.

Churning of cream and subsequent working of butter cause rupture and deformation of numerous fat globules. The deformed globules then release their crystalline fat which becomes embedded in the continuous liquid fat phase. The crystalline fat comprises the major solid portion of butter. Polarized light photomicrographs show that churned butter contains small crystals evenly distributed throughout the product (Vyshemirskii, 1965). Considerable crystalline fat also remains trapped within the intact globules (King, 1964).

Electron microscopy using the replica technique was applied by Okada (1962) to the study of butter preparations. Rhombic crystals were reported present in the butter surface. As the observations were limited to the surface of the butter, little useful information could be obtained regarding the structure of the substance as a whole.

Another view concerning butter structure was presented by Knoop and Samhammer (1962). They felt that polarized light microscopy did not reveal correct information concerning the amount and location of crystalline fat in butter. Using X-ray methods, they found that less than 20% of the fat in butter was in the crystalline form at 18-20^{o}C. Conversely, King (1955) suggested that conventional butter can contain 50% of more crystalline fat at 20^{o}C.

It was further proposed by Knoop and Samhammer that milk fat existed in an amorphous form which was presumably liquid. Only trisaturated and monosaturated triglycerides were thought to contribute to the crystalline fraction of butter. Butter was considered as a liquid of high viscosity rather than as a solid.

The theory of Knoop and his co-workers was supported by electron micrographs of slices of butter exposed for six months to OsO_4 before

preparation as 30 mμ sections (Knoop and Wortmann, 1962). No intact fat globules were observed using this technique. Rather, fat "micro-granules" were described. The microgranules were thought to be derived from damaged fat globules. Wortmann (1963) reported that only remnants of the original fat globule membrane could be seen using electron microscopy. The granular structure was especially evident in churned butter and was found to be influenced by cream cooling treatment. A homogeneous structure was also proposed, occurring most noticeably in continuous butter. Knoop (1964) indicated that much butter appeared to be between the two structural extremes.

2. Crystallization of Milk Fat

a. Crystallization of Pure Milk Fat

Studies involving pure milk fat are extremely useful in clarifying the mode of milk fat crystallization in cream. Milk fat contains a large number of fatty acids arranged as triglycerides. If molten milk fat is cooled rapidly, a large number of small crystals forms. Slow cooling produces fewer large crystals (Wiley, 1946). The crystals formed on slow cooling consist of layers of lower melting glycerides built upon a nucleus of high melting glycerides. Rapidly cooled milk fat contains 'mixed crystals' composed of dissimilar glycerides; the crystals do not exhibit laminar structure (Mulder, 1953).

There is more solid fat in rapidly cooled milk fat than in slowly cooled milk fat (de Man, 1963a). Stepwise cooling usually results in a reduction of the amount of solid fat because progressively fewer glyceride fractions are available for nucleation as cooling proceeds. The temper-

ature history of milk fat can also greatly influence the solid fat content, especially in the case of stepwise-cooled fat (Vasic and de Man, 1965b).

It is thus evident that both size and quantity of crystals affect the solid fat content and hardness of milk fat. Crystal composition also influences the solid fat to liquid fat ratio. In addition, seasonal variations in the proportions of unsaturated and short-chain saturated fatty acids in the glycerides of milk fat govern the solidification of the fat. Solid fat content is higher in winter milk fat than in summer fat as a result of the increased content of higher melting glycerides in winter fat (de Man, 1961a; 1961b).

b. Crystallization of Milk Fat in Cream

(i) Effect of Cooling Rate

In pure milk fat preparations, the fat phase is continuous. The effects of changes in crystallization behavior of the fat can thus be quickly and readily measured by such techniques as dilatometry, calorimetry and differential thermal analysis. In cream, however, the fat is dispersed in globular form and a longer time is required for crystallization to occur. The fat exhibits a 'time lag' in attaining equilibrium throughout the globule (Brunner and Jack, 1950).

If cream is cooled rapidly from pasteurizing temperatures, the amount of solid, crystalline fat is at a maximum. Slow cooling of cream results in a reduction in the amount of crystalline fat. There is consequently an increase in the liquid fat content of the cream. Butter made from rapidly cooled cream is harder than that from slowly-cooled cream (Coulter and Combs, 1936; Dolby, 1941c, 1954; Huebner and Thomsen, 1957b; de Man and Wood, 1959c).

The globules of milk fat in cream impose a physical limitation on the size of crystals which can be formed when cream is cooled. The crystalline milk fat occupies only a part of the total globule volume and crystals tend to be very small. However, rapid cooling of cream can produce smaller crystals than would a slower rate of cooling. Some unusually large crystals can also result when cream is cooled rapidly (de Man and Wood, 1959a).

(ii) Effect of Dispersion of Fat Globules

The degree of dispersion of fat globules affects crystal size (de Man, 1964b). It is therefore possible that, since dispersion of globules by homogenizing cream reduces globule size, smaller crystals could be formed in cooled homogenized cream. Homogenized cream has been found to require a longer time to reach thermal equilibrium after cooling than does unhomogenized cream (Mulder, 1942; Phipps, 1957).

(iii) Influence of Cooling and Holding Temperatures

Several factors influence the size, number and type of fat crystals in cooled cream. The rate of cooling has considerable effect. In addition, cream cooling temperatures and holding temperatures affect crystallization of milk fat.

Cooling and holding at low temperatures cause an increase in the crystalline fat content of cream. Higher holding temperatures or less thorough cooling will result in increased liquid fat. Storgårds (1938) noted that cream cooled to 14°C yielded soft butter while hard butter was obtained by cooling cream to 8-9°C. Similar results have been reported by several other workers (Coulter and Combs, 1936; Mulder, 1953; King, 1953; Dolby, 1954, 1959).

Tempering has also been found to alter crystallization of milk fat in cream. If cream is cooled to a low temperature and held there for several hours, then rewarmed and held at a high temperature for several hours, and finally cooled to the churning temperature, the crystalline fat content is reduced. The Alnarp method based on this principle was developed in Sweden for ripened cream butter (Samuelsson and Pettersson, 1937; Adriani and Tamsma, 1946; Fisker and Jansen, 1962; Johansson and Swartling, 1963). The procedure resulted in decreased butter hardness. Olsson (1948) pointed out that the treatment was most effective when Iodine values of milk fat were between 27 and 31. For values above or below this range, the treatment was much less effective in reducing the solid fat content of cream.

Mulder (1953) explained that warming of cooled cream caused partial melting of the solid fat. The melted fat then combined with the residual solid portion and re-crystallized at a higher temperature. When the cream was finally cooled, fewer mixed crystals formed and a lower solid fat content was attained.

Adaptation of a modified Alnarp procedure for sweet cream pasteurized in a Vacreator was reported by Dolby (1954, 1959). Precooling of cream at 45-48^{O}F was not in itself effective as a means of lowering butter hardness. However, a short precooling period of 8 seconds at 45-48^{O}F effectively limited fat losses in buttermilk. The actual hardness reduction was achieved by holding of the cream at 65-66^{O}F for one-half hour to one hour. It was also found that holding of cream at 65^{O}F without precooling resulted in decreased butter hardness although fat losses in buttermilk were high. In a recent study, Wood and Dolby (1965) noted that a permanent reduction in butter hardness could be obtained using the modified precool-

ing procedure for plate-pasteurized cream.

(iv) Effect of Seasonal Variations in Milk Fat Composition

Crystallization of milk fat in cream is greatly affected by seasonal variations in the content of unsaturated and short-chain fatty acids in the fat. These differences are related to the feed of the cow. In the winter months, fewer unsaturated and short-chain fatty acids are present than in the summer months.

Winter milk fat has been found to contain more solid fat when cooled than does summer milk fat. Mulder and Klomp (1956) pointed out that, at 18°C, 50% of winter milk fat was solid, whereas only 35% of summer fat was in the solid state. The solid fat content of cream directly affects the hardness of butter. Dolby (1954) attributed 80% of variations in butter hardness to seasonal changes in milk fat composition.

3. Hardness of Butter and the Setting Phenomenon

a. Hardness of Butter

Hardness or firmness may be described as the property of a substance which enables it to resist deformation. Hardness of plastic fats such as butter depends largely on the proportions of solid fat and liquid fat in the product. The three-dimensional structure formed by the fat crystals imparts rigidity and hardness to the butter (van den Tempel, 1958). Spreadability is another term synonymous with hardness (de Man, 1961c).

Butter is a plastic substance and exhibits a yield value below which butter acts as a solid; it will not flow until the yield stress is exceeded. Such plastic-disperse systems possess elastic properties at

small deformations (van den Tempel, 1961).

b. Setting of Butter

The continuing increase in hardness of butter over a period of time following manufacture is termed 'setting'. Setting is a continually changing property whereas hardness is a 'static' one, representing a condition prevailing at one particular time. A series of instrumental hardness measurements is used in following the setting pattern of butter. The measurements are made at regular intervals under controlled conditions. The temperature of measurement is particularly important and must be constant if accurate comparisons of treatments are to be made.

The occurrence of setting in conventional butter was noted by Wode (1934). Storgårds (1938) indicated that hardness of butter decreased on working but increased again when butter was allowed to stand for a time. The hardness increase was attributed to crystallization of liquid fat released during working. Mulder (1949) remarked that the setting phenomenon warranted attention since the hardness of butter could increase as much as twenty times after completion of working. Huebner and Thomsen (1957b) noted the importance of setting in determining the hardness of butter. Much valuable information on setting of both conventional and continuous butters has been contributed by de Man and Wood (1958a; 1959c).

4. Mechanism of Setting

The crystalline fat in butter influences both hardness and the related property of setting. Conventional butter contains numerous crystals of colloidal dimensions embedded in the liquid free fat phase. According to King (1964), these small crystals are free to move and can form a network. Crystals trapped in the intact fat globules of butter cannot par-

ticipate in the formation of a network. Other larger crystals are also capable of forming a structure but they do not possess freedom of movement (de Man and Wood, 1958b).

Setting of butter has been attributed to the crystallization of undercooled fat (Storgårds, 1938; Baron, 1952). However, it was found that butter underwent setting in the absence of undercooled fat. King (1953) stated that cream cooled to a low temperature contained no undercooled fat. He also pointed out that a framework built up by crystallization of undercooled fat would break down irreversibly on reworking. Conventional butter, on the other hand, partially regained its hardness after reworking.

Other workers felt that polymorphic transformations of crystalline fat in butter affected setting. Tverdokhleb (1962) considered that the phase changes in milk fat were very important in determining the structure of butter. Holding of cream at 15-20°C was claimed to increase the stability of crystalline modifications. In turn, these stable crystal forms were believed to cause harder butter. Brunner and Jack (1950) stated that the fat in cream was in equilibrium after eighteen hours so that the influence of polymorphism after churning was unlikely. In reviewing the subject of polymorphism in milk fat, de Man (1963b) could find no clear indication of any direct effect of polymorphism on the rheological properties of butter.

Thixotropy has been proposed by some investigators to be the main cause of setting in conventional butter (Mulder, 1949; Huebner and Thomsen, 1957b; de Man and Wood, 1958a, 1958b). Evidence for the occurrence of thixotropy is found in the behavior of conventional butter following

the completion of working. There is no continuing crystallization in conventional butter since its temperature does not increase after working ceases (de Man and Wood, 1958a; Kacherauskis and Motekaĭtis, 1964).

The number and size of crystals present in conventional butter strongly indicate the possibility of thixotropic rearrangement. The influence of temperature on setting can also be explained in relation to thixotropy. McDowall (1962) stated that the interlocking of crystals appeared to be the most probable cause of setting in churned butter.

5. Factors Affecting Hardness and Setting of Butter

Both hardness and setting of churned butter are influenced by the crystalline fat content of the product which is, in turn, determined largely by the state of the crystalline fat in cream. Seasonal differences in milk fat composition exert a major effect on butter hardness (de Man and Wood, 1958a). Hardness can also be altered substantially by changes in temperature treatment of the cream. Variations in other stages of the buttermaking process or changes in non-fat constituents have much less influence on the hardness of the product (Dolby, 1941c). For instance, de Man and Wood (1959d) reported that increases in moisture and gas contents of butter tended to cause a slight but definite lowering of butter hardness. Thomsen (1955) agreed that the rate of cream cooling was a primary factor in determining hardness of butter. Mechanical working of butter and conditions of storage can also change its hardness and setting (de Man and Wood, 1959c).

Wood (1964) stated that setting of butter was influenced by seasonal variations in the hardness of milk fat, by the cooling treatment of cream and by the method of pasteurizing cream. Summer butter, which

is initially softer than winter butter, sets to lower hardness values. Printing of butter reduces its initial hardness and thereby limits the extent of setting. If cream for buttermaking is rapidly cooled, the butter is initially harder and sets to higher hardness values than does butter from slowly cooled cream (de Man and Wood, 1959c). Tempering of cream and Alnarp treatment have been shown to lower the hardness of butter and to reduce setting, depending on the method of pasteurizing cream (Wood and Dolby, 1965).

Butter from Vacreator-pasteurized cream sets very rapidly to high hardness values; butter from plate-pasteurized cream sets more slowly and attains lower hardness after five hours. An increase in the steam intensity of Vacreator treatment causes a rise in the rate of setting. Differences in butter hardness do not persist, as all butters exhibit similar hardness after several days' storage at $45^{o}F$ (Wood and Dolby, 1965).

Since the initiation of the present study, Dolby (1965) has suggested that homogenizing of cream can affect the hardness and setting of butter. Some relationship may exist between the effects of Vacreation and those of homogenizing. Both treatments cause a substantial increase in the number of small fat globules in cream. Vacreation also produces a small rise in the number of globules with diameter greater than 10μ (Dolby, 1953).

The temperature at which butter is stored after manufacture is an important factor influencing hardness and setting. Setting is quite rapid at $12\text{-}14^{o}C$ (Storgårds, 1938). Huebner and Thomsen (1957b) reported a reversal of setting by cold storage at $-6^{o}F$. Other workers

stated that freezing of butter at -20°C before it had completely set interrupted the setting process (de Man and Wood, 1959c). Setting resumed normally when the butter was tempered at 4-5°C for two weeks. The solidification of liquid free fat in butter was believed to prevent setting at low temperatures.

Changes in churning temperatures and in conditions of washing and working of butter can also affect hardness. Mulder (1947) reported that churning at 9°C or at 12°C produced harder butter than did churning at 15°C, although the hardness differences were not large. Use of low temperature wash water and low working temperatures have been claimed to cause a decrease in butter hardness (Coulter and Combs, 1936; Thomsen, 1955). However, changes in temperatures of churning and washing have a minor effect in comparison with seasonal variations and cream cooling conditions (Dolby, 1941c; 1959).

6. <u>The Thixotropic Phenomenon</u>

a. <u>Characteristics of Thixotropic Systems</u>

Thixotropy may be defined as the isothermal, reversible sol-gel transformation undergone by some disperse systems when work is performed on them. Freundlich (1935) stated that the phenomenon depended on the presence of attractive and repulsive forces between the particles of the disperse phase. The forces of attraction are probably van der Waal's forces; the nature of the repulsive forces is not clearly understood (van den Tempel, 1963).

There are several requirements for thixotropy in a two-phase system. The particles of the disperse phase must be small, numerous and anisometric. Needles, rods, and plate-like particles are commonly

thixotropic. Thixotropy is not usual in dispersions of spherical particles (Pryce-Jones, 1936). Thixotropic substances must contain a certain proportion of particles with diameter 1 μ or less. A limiting size of 5 μ has been suggested (Freundlich, 1935).

Thixotropic changes are isothermal as well as reversible. No temperature change is required to promote the sol-gel transformation. The gel system breaks down to form a sol when sufficient disturbing force is applied. If the system is allowed to stand, the gel reforms. Reformation of the gel is not instantaneous for a truly thixotropic substance. Pryce-Jones (1936) stated that the time which elapsed before the sol reverted to the gel state distinguished thixotropy from the related property of plasticity. Complete reformation of the gel is not necessary; partial gelation is considered indicative of a thixotropic change (Freundlich, 1935).

b. Measurement of Thixotropic Changes

A single-point method is a simple means of detecting the occurrence of thixotropic breakdown and rebuilding. The time for a gel to reform after a suspension is shaken in a tube can be regarded as a criterion of the degree of thixotropy present (Freundlich, 1935). This type of measurement is arbitrary and depends on conditions such as the size of tube, volume of substance and length of time of agitation. Goodeve (1939) suggested a single-consistency curve and defined a 'coefficient of thixotropy'. A weakness of the single-consistency curve was that no indication was given of the original state of the material. Determinations of consistency were made only after completion of shear.

Green and Weltmann (1946b) reviewed methods of measuring thixotropy. They felt that single consistency curves were not useful for study-

ing changes in thixotropic systems. The only method recommended was a 'double consistency curve', because the state of the substance before shear was considered as important as its state after shear. If there was no difference in consistency, the substance was regarded as non-thixotropic. A decrease in consistency during shear, followed by an increase in consistency on standing after shear indicated a thixotropic substance. Duration of shear should also be considered because thixotropy is a time-dependent phenomenon (Green and Weltmann, 1946a).

c. Relation of Thixotropy to Plasticity

A plastic material is one which exhibits a yield value. The substance will not flow at stresses below its yield stress. At stress values greater than the yield stress, the substance does flow. A plastic substance is thus non-Newtonian in its rheological behaviour; the rate of flow is not proportional to the shear applied. Röder (1939) stated that a plastic would retain the shape it adopted during moulding, even after the stress had ceased to act.

It is notable that many thixotropic systems are plastics, being dispersions of solid particles in a liquid continuous phase (Scott-Blair, 1938a). One must differentiate between plasticity and thixotropy. Plasticity is not reversible and deformation is non-recoverable. Thixotropy is reversible; rebuilding of the structure occurs after shear ceases.

d. Examples of Thixotropic Systems

Among plastic substances exhibiting thixotropy are gypsum pastes (Freundlich and Juliusburger, 1935), bentonite, (Freundlich, 1935; Röder, 1939), paints and printing inks (Green, 1942) and Heather honey (Pryce-Jones, 1942). Some materials which are not considered as plastics have

also been reported to show thixotropy under limited conditions. Weltmann (1943) stated that linseed oil acted as a thixotropic plastic at certain rates of shear.

Studies with model systems of 20-30% concentrations of glyceryl tristearate crystals in paraffin oil have shown that small, needle-shaped crystals can undergo thixotropic alterations (van den Tempel, 1958; Nederveen, 1963). The crystals formed a three-dimensional network in the continuous oil phase. Recovery of original hardness after working was incomplete because of the breaking of some irreversible bonds during shear (van den Tempel, 1961). It was also noted that samples of undisturbed milk fat did not exhibit setting whereas samples which had been mechanically worked increased in hardness over a period of fourteen days (de Man, 1962).

3. Thixotropy and Setting of Conventional Butter

Conventional butter is an unusual example of a thixotropic substance. Unlike other two-phase systems, butter has interchangeable phases consisting of solid fat and liquid fat. Butter contains numerous small elongated crystals which are capable of interlocking to form a 'scaffolding' (de Mann and Wood, 1958b). Working of butter causes the crystals to become disoriented and the structure is partially destroyed. Butter is thus soft when freshly made. When it is allowed to stand under isothermal conditions, its hardness increases.

Reworking of butter which has become completely set causes a permanent hardness loss (Mulder, 1949). Partial regain of butter hardness after reworking can be attributed to the presence of two types of crystal structure. Large crystals form a primary structure with irreversible bonds. Once these bonds are broken by shear, they cannot be reformed. On the

other hand, very small crystals are capable of forming a thixotropic structure which can be rebuilt after rupture (de Man and Wood, 1958b; van den Tempel, 1958).

There are numerous factors which can affect thixotropic setting of butter. The prevailing temperature is important because small crystals require mobility in order that rearrangements can occur. At low temperatures, such as those of freezing, the liquid phase of butter is solidified and crystals cannot move (de Man and Wood, 1959c). Setting of butter is thereby halted. If butter is tempered following freezing, setting resumes as crystals again become mobile. High temperatures also prevent setting by causing melting of some crystals so that insufficient particles remain to form a network.

Processes which alter the ratio of liquid fat to solid fat in butter also influence setting. Slow cooling or stepwise cooling of cream reduce the amount of crystalline fat. Butter from such cream sets to lower hardness values than does butter from rapidly cooled cream. In addition, any process which changes the size distribution of crystals in butter is capable of altering the setting pattern. Wood and Dolby (1965) found that butter from Vacreated cream set very markedly and contained a large quantity of small fat crystals. Homogenizing of cream may have a similar effect.

7. Measurement of the Hardness of Butter

a. Instrumentation

One of the most important rheological properties of butter is hardness or firmness. In measurements of hardness of plastic fats, the rate of shear must be specified. Shear rate influences the resistance

of butter to deformation (Dolby, 1941b). The temperature of measurement should also be stated. McDowall (1953) has reviewed methods of hardness measurement applied to butter. These include penetration, sectility, extrusion, viscosity and various spreadability techniques.

Penetrometers of various types have been developed. The cone penetrometer records the depth of penetration of a metal cone into a sample at a given temperature. The time of penetration is usually five seconds (Rich, 1942). The measurement indicates a yield value which makes it suitable for plastic substances (Haighton, 1959). Cameron (1945) noted that measurements with a cone penetrometer are rapid enough to be within the relaxation time of the test material. A disc penetrometer, used by Kruisheer and den Herder (1939), also measures a yield value. Modifications to this instrument have been made by de Man and Wood (1958a). Small samples are used and control of temperature can thus be readily achieved. If the depth of penetration and rate of shear are constant, the force needed to cause the disc to penetrate the sample can be used as a measure of hardness. The force is expressed as a function of the area of the disc surface.

The sectility method of Dolby (1941a) involved use of a wire to cut through a sample of butter. A 'practical yield value' was obtained by measuring the minimum load which, when applied to the wire, produced a measurable flow. The load was found to be proportional to the diameter of the wire and to the length of the wire contacting the sample. The disc penetration apparatus of de Man and Wood (1958a) was also adapted for sectility measurements.

Prentice (1954) determined spreadability of butter by extruding

a cylinder of butter at a constant rate through a small orifice. He felt that the yield value was not as important as the resistance of the butter to 'rapid deformation'. The minimum thrust of the piston at the moment the sample cylinder was emptied was regarded as a measure of resistance to extrusion. The values obtained were related to the spreadability of the butters tested. Dixon (1964) used the same instrument in spreadability tests.

Several workers have devised instruments to measure the spreadability of butter. One such device consisted of a knife-blade and a wire which were drawn by a motor over the butter surface at a specified depth. The blade was used to estimate spreadability while wire was used in hardness measurements (Huebner and Thomsen, 1957a). Spreadability was considered to be a complex property of which brittleness and hardness were the most important elements. Riel (1960) and Kapsalis <u>et</u> <u>al</u>. (1960) made slight changes in the spreadability instrument. It was claimed to be more sensitive than extrusion methods.

Davis (1937) and Scott-Blair (1938b) recommended use of a rheometer which measured the viscosity of butter at varying stress values. The viscosity, expressed in absolute units, was greatly dependent upon the temperature of measurement. Viscosity values obtained were found to be useful in estimating the spreading capacity of butter samples.

Wood and Dolby (1965) modified the Prentice extruder for penetration measurements by means of an adapter to hold round sample frames. A shaft with penetration disc fits onto the drive shaft of the instrument. The same adapter can be used for sectility measurements, by substituting slotted frames and a shaft with a wire cutter.

b. Correlation of Methods of Measuring Butter Hardness

One of the major problems in interpreting the results from instrumental measurements of butter hardness is the lack of common units and standard test procedures. Thus it is not always possible to compare the results obtained by one method with those from another method. Use of different measuring temperatures can account for considerable variation in results. Shear rate, sample size and conditions imposed by instruments differ from method to method.

A few workers have attempted to relate an instrumental method to subjective assessments of the rheological properties of butter. Kruisheer and den Herder (1939) reported that penetrometer hardness measurements were in close agreement with graders' scores for firmness of butter. No analysis of the relationship was made. Prentice (1954) equated extruder thrust values with spreadability scores obtained by a trained panel. For 300 samples, a significant correlation coefficient of 0.896 at the 1% level indicated a positive relationship. In another study, Huebner and Thomsen (1957a) compared eight sets of consumer scores for spreadability of butter with instrumental spreadability readings. A correlation coefficient of -0.60 at the 1% level was obtained. Kapsalis et al.(1960), reported that spreadability and consumer panel ratings for 109 samples were related with correlation coefficient of -0.757 at the 1% level. A coefficient of -0.6767 was obtained between hardness values measured with a wire and panel scores. Riel (1960), in a study of 57 butter samples, compared panel scores to spreadability values and obtained a correlation coefficient of 0.94, significant at the 1% level.

Some attempts have been made to eliminate subjective scoring

and to relate results of various objective methods of assessing butter hardness and spreadability. Early work by Dolby (1941a) showed that the Scott-Blair rheometer and the sectility apparatus measured the same property of butter. Fifty samples tested yielded a linear relationship. The temperature of measurement was not specified for this study. Huebner and Thomsen (1957a) and Kapsalis _et al_ . (1960) compared mechanical spreadability and hardness determinations. Both measurements were made with the same instrument. Huebner and Thomsen reported a correlation coefficient of 0.95, significant at better than 1% level. A slightly lower coefficient was obtained by Kapsalis _et al_. (1960). A measuring temperature of 55.5°F was used in both studies. Penetrometer and sectility values were compared by de Man and Wood (1958a). A highly significant correlation coefficient was found between penetrometer values measured at 17°C and sectility measurements at 12°C. For conventional butter the coefficient was 0.98, significant at the 1% level.

III. EXPERIMENTAL METHODS

Pilot-plant equipment for semi-commercial butter manufacture was not available for cooling and holding of cream with a sufficiently accurate degree of temperature control. In addition, the desired small quantities of butter could not be made readily using churns of large capacity. Thus small-scale holding equipment was constructed for this project and a laboratory churning method was devised. In this way, cooling, holding and churning temperatures could be closely regulated and a large number of churnings could be made in the limited time available.

1. Cream Processing

Special or Number One Grade cream was obtained from the bulked supply of a local creamery. The fat content of the cream ranged from 33% to 37%. The cream was neutralized to a titratable acidity of 0.10% with a sesqui-carbonate neutralizer.

The cream was pasteurized in a Saf-Gard home pasteurizer of two-gallon capacity, with hot water as the heatant. The neutralizer was added when the cream temperature reached 80 - 90°F. The cream was heated to 170°F and cooled after holding for ten minutes. Approximately ten pounds of cream were pasteurized for each churning.

2. Homogenizing of Cream

A single-stage Gaulin laboratory homogenizer was used for homogenizing cream. Pressures in the range of 1000-1200 psi were found to cause undue extension of the churning time. Consequently, a pressure of 800 psi was used in all trials involving homogenizing of cream.

The temperature of the cream at the time of homogenizing varied with the cooling treatment to be used. In all cases, the cream temperature was over 100°F. The homogenizer was connected directly to the cooling coil when rapid cooling of cream followed homogenizing.

3. Cooling of Cream

Unless otherwise indicated, the cream for all churnings received a preliminary cooling to 100°F by circulation of tap water at 60-65°F through the pasteurizer. The method of cooling below 100°F depended on the particular treatment used.

a. Slow Cooling

For slow rates of cooling, the cream was cooled with tap water in the pasteurizer from 100°F to 60-65°F. This stage of cooling required about thirty minutes. Further cooling was accomplished by placing the cream in a water bath at 45-50°F and stirring it until the desired final temperature was reached. The rate of temperature decrease in this period was approximately 2°F in five minutes.

b. Rapid Cooling

Rapid cooling of unhomogenized cream was carried out by circulating the cream through the cooling coil with the homogenizer at zero gauge pressure to maintain a rapid flow rate. Cooling water at 34-36°F was recirculated through the coil which consisted of a double tube heat exchanger with an inner stainless steel tube 24.75 feet long and 0.625 inch O.D. Homogenized cream was cooled in a similar manner except that a gauge pressure of 800 psi was used.

The rate of cooling was calculated from the following equation:

$$\frac{\Delta T \times S \times \rho \times V}{t} = \text{heat loss/unit time}$$

where V = volume of cream (cc) pumped in t seconds

ρ = density of cream (g/cc)

S = specific heat of cream (g-cal/gram)

ΔT = initial temperature of cream - final temperature of cream ($^{\circ}$C)

t = time in seconds required to pump volume V

A specific heat value of 0.72 g-cal/gram $^{\circ}$C was used in the calculation, based on the mean fat content of the creams tested (Charm, 1963). An average figure of 1.00 was used for density of cream (Macintire, 1937). The cream temperature was measured with a mercury thermometer both before and after cooling. A measured volume of cream was collected from the outlet of the coil; the flow time was obtained with a stop-watch.

The cooling rate data for ten representative lots of homogenized and unhomogenized creams is summarized in Table 1. The rate of cooling in the coil varied with the initial temperature of the cream and with the temperature of the cooling water. The flow rate of the cream was found to be slower when pressure was applied to the homogenizing valve than when no pressure was used.

c. Precooling and Stepwise Cooling

The method was adapted from a procedure of Wood and Dolby (1965). Three variations were used with both homogenized and unhomogenized creams in summer and winter seasons: a control treatment, a precooled treatment and a stepwise-cooled treatment. The procedure for winter cream is detailed here; summer cream treatments were identical except for the final holding and churning temperature.

(i) Control Treatment

In winter, the control cream was cooled rapidly to 50°F, held overnight and churned at that temperature. This treatment is hereafter designated as 50-50-50.

(ii) Precooled Treatment

Winter cream was cooled rapidly to 40°F and held for two hours. Next, the cream was warmed to 65°F, using tap water not exceeding 78°F, and held at this temperature for three hours before cooling slowly to 50°F, holding overnight and churning at 50°F. The designation used for this treatment is 40-65-50.

TABLE I COOLING RATE DATA FOR 10 REPRESENTATIVE LOTS OF CREAM

	UNHOMOGENIZED CREAM	HOMOGENIZED CREAM
MEAN COOLING TIME FOR 1000 CC	56.5 SEC	65.7 SEC
MEAN COOLING RATE CALCULATED	544.5 G-CAL/SEC	511.1 G-CAL/SEC

(iii) Stepwise-Cooled Treatment

Stepwise-cooled winter cream was initially cooled rapidly to 65°F and held for three hours. The cream was then cooled slowly to the churning temperature of 50°F and held overnight at that temperature. The stepwise cooling treatment is designated as 65-50-50.

In summer months, the same sequences were used, with overnight holding and churning temperatures of 45°F rather than 50°F. Use of a lower temperature resulted in summer butter which was not excessively sticky.

4. Holding of Cream

The cooled cream was held at the required temperature in the series of two-gallon jacketed vats illustrated in Figure 1. The vats permitted maintenance of the cream temperature by circulation of refrigerated water through the jackets. The cream temperature was thermostatically controlled by means of an arbitrary-set controller with an accuracy of $\pm$ 0.1°F. The controller probe was immersed in the cream and agitation was provided by motor-driven stirrers.

The cooled cream was generally held for 16-18 hours before churning; holding was at 45°F in summer and at 50°F in winter. The only exception was in experiments involving holding of cream for one or two hours at 40°F followed by immediate churning.

Holding for three hours at 65°F in the precooling and stepwise cooling treatments was accomplished by placing the cream container in a water bath at 65°F .

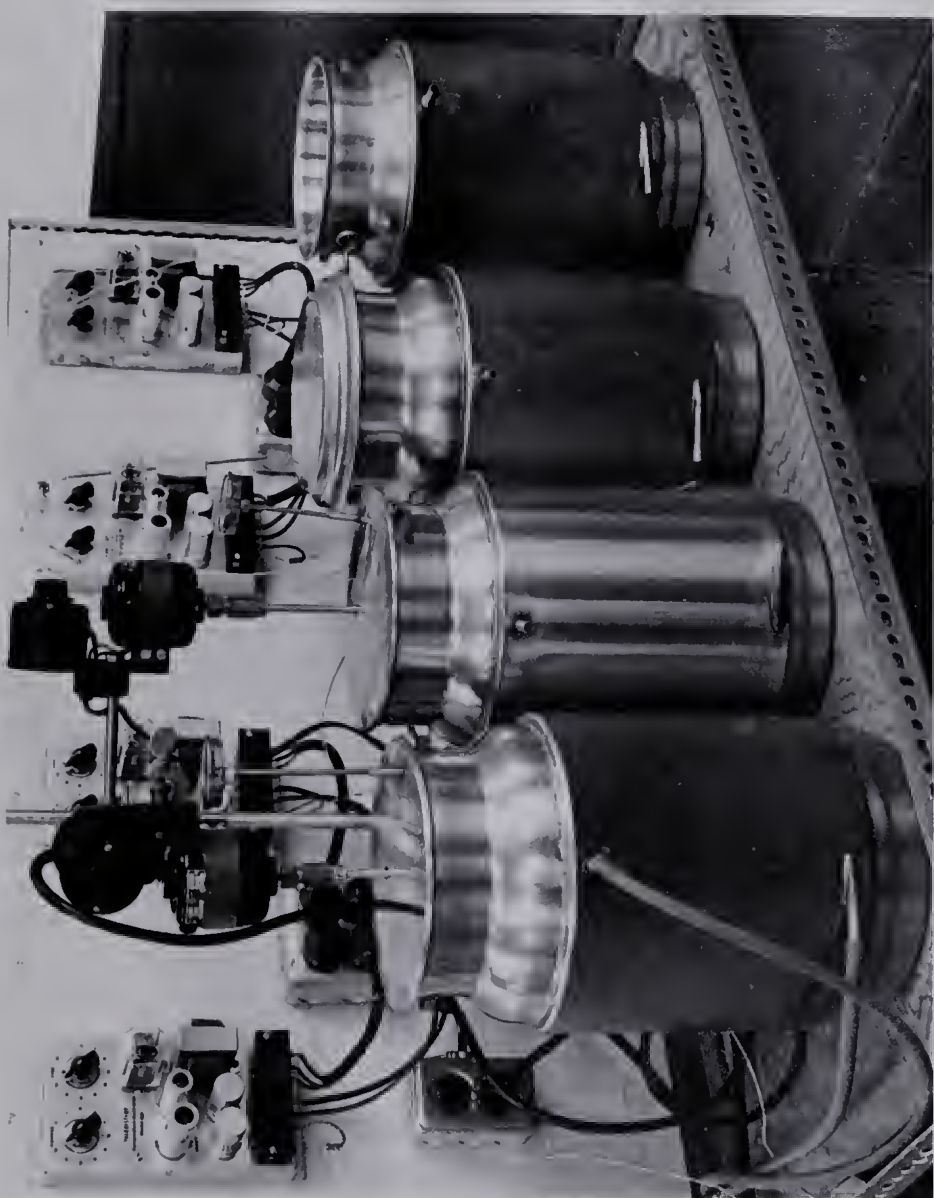


FIGURE I CREAM HOLDING VATS

5/8/11-90

5. Addition of Liquid Fat to Cream

The fat for addition to cream was obtained from butter of the current month. The butter was melted, cooled, and the clear oil was decanted carefully from the curd. The required amounts of oil were calculated on the basis of the fat content of creams used.

The liquid fat was added to unhomogenized cream just before the cream was cooled rapidly in the coil. For homogenized cream, the liquid fat was added after homogenizing but before rapid cooling. The temperature of the fat was the same as the cream temperature. Cream for the control churnings did not contain added fat and was cooled rapidly to the churning temperature. All creams were held overnight before churning.

6. Churning, Working, and Sampling Butter

All experimental butters were churned in a Taylor ice cream freezer, Model 430 with a capacity of 1.20 Imperial gallons, modified by removal of the scraper blades. The speed of the beater was 220 rpm. Because of the limited capacity of the freezer, the cream for each churning was divided into three lots of approximately three pounds; each lot was churned separately. The granules and buttermilk from the first and second lots were held at 45^{o}F until the third lot was completed. The churning time for each lot ranged from 5 minutes to 30 minutes, depending on the season.

After completion of churning, the buttermilk was drained from the granules. Washing of granules with water at 45°F for three minutes was followed by draining and manual working with wooden paddles in a room maintained at 40°F. The temperatures of all butters, measured immediately after the completion of working, were in the range of 49-52°F.

As soon as working was completed, the butter was shaped into a flat slab and samples were cut with cylindrical frames made from 2.9 cm lengths of 0.5 cm O.D. stainless steel tubing. The samples for five hours setting were placed in a water bath maintained thermostatically at 55.5°F with a controller having an accuracy of ± 0.5°F. Four additional samples were taken from each churning, wrapped in plastic film to prevent desiccation and stored at 40°F for hardness measurements at one week and four week intervals.

7. <u>Determination of Hardness and Setting of Butter</u>

Hardness of butter was measured with the Kruisheer and den Herder disc penetrometer, using the method as modified by de Man and Wood (1958a). The accuracy of the penetrometer was determined for both summer and winter butters. From the results presented in Table 2, it appears that the penetrometer accuracy is exceptionally high.

TABLE 2 ACCURACY OF HARDNESS MEASUREMENTS WITH THE MODIFIED KRUISHEER AND DEN HERDER PENETROMETER *

	SUMMER BUTTER	WINTER BUTTER
RANGE OF HARDNESS VALUES [$kg/4\ cm^2$]	4.95–5.20	6.55–6.60
MEAN	5.05	6.59
AVERAGE DEVIATION FROM MEAN	± 1.54%	± 0.28%

* 10 REPLICATES OF EACH BUTTER WERE TESTED

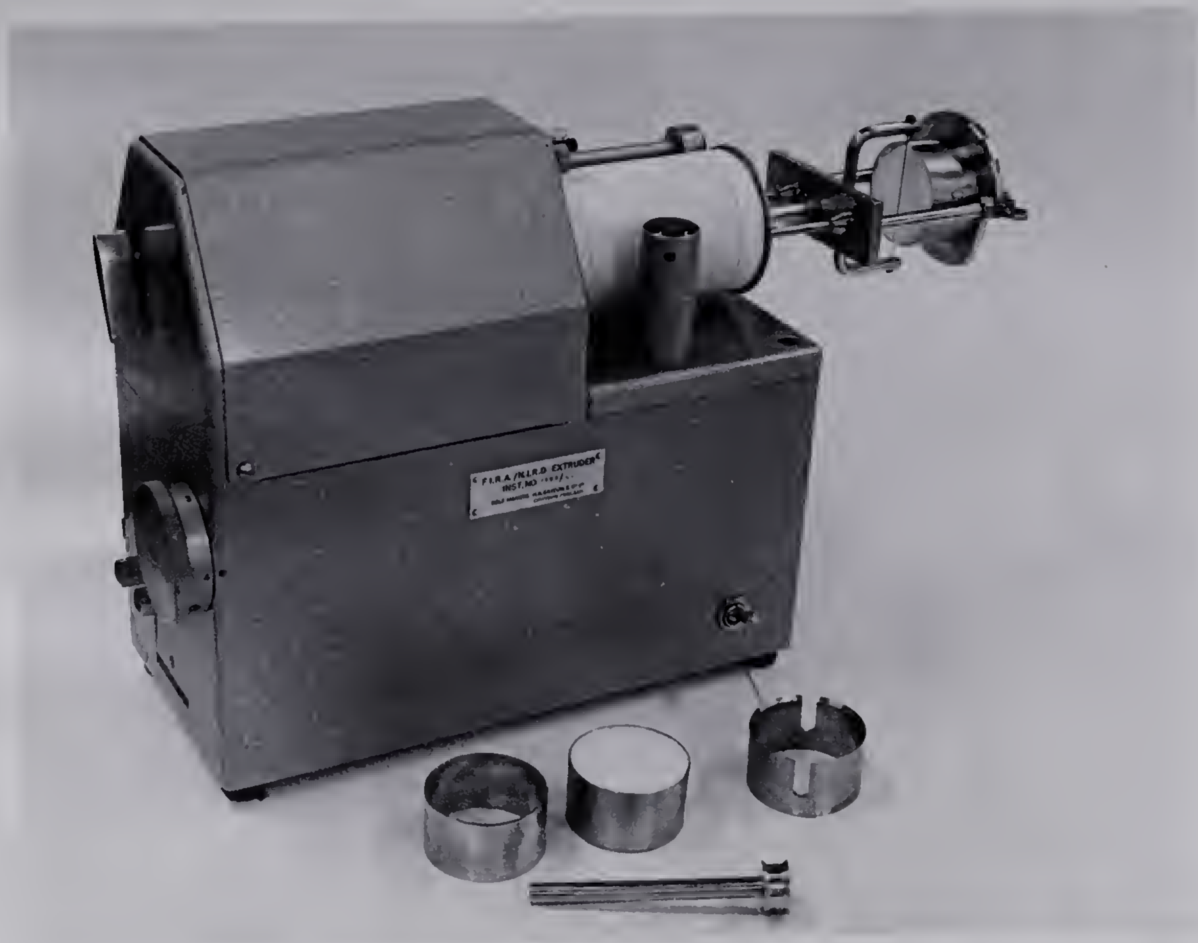
In determination of the setting pattern of butter, duplicate samples were tested for hardness at completion of working and at half-hour intervals for five hours thereafter. The samples were stored at 55.5°F during this time; measurements were made in a room maintained at 40°F. The samples stored at 40°F were tempered for 24 hours at 55.5°F before determining hardness.

In a few instances, the hardness of the 30-day samples exceeded the 10 kilogram capacity of the disc penetrometer and the modified NIRD extruder with penetration attachment was substituted (Figure 2). This instrument had a capacity of 12 kilograms and a shear rate of 1.56 inch/minute.

8. Moisture Content of Butter

Because a manual procedure was used in working the experimental butters, it was difficult to control the moisture content. The range of moisture content for 50 butters tested in duplicate by the modified Kohman method was 14.5-17.7%; the mean was 15.95%.

Although the range was considerably greater than that normally found in commercial butter, it is unlikely that variations of this magnitude would unduly influence the hardness or setting of butters. Moisture has been found to have a small but definite effect on butter hardness (de Man and Wood, 1959d).



<u>FIGURE 2</u> NIRD EXTRUDER MODIFIED FOR PENETRATION AND SECTILITY MEASUREMENTS

9. Polarized Light Microscopy

A dilution method was used for the microscopic examination and preparation of samples for photomicrographs, based on a procedure developed by de Man and Wood (1959a). The butters were allowed to set for at least seven days. A small quantity of butter was weighed accurately onto a clean, dry slide. An equal quantity of paraffin oil was then weighed onto the same slide. The butter and oil were mixed very thoroughly with a small spatula. Weighing and mixing were done in a room maintained at 40^{o}F.

A small amount of the butter-paraffin oil mixture was transferred to a special slide. An indentation in the slide allowed a layer of the mixture exactly 10 microns thick to be formed when the mixture was pressed with a cover slip.

The prepared slides were observed under polarized light with a Zeiss-Winkel standard polarizing microscope, having illumination built into the base. A Zeiss camera with adapter was used for photomicrographs. The magnification on all photomicrographs was 500 x.

IV. RESULTS

1. Homogenizing of Cream

In preliminary experiments, cream was homogenized at $100^{o}F$, $140^{o}F$ and $170^{o}F$. The results, shown in Figure 3, indicate that, within this temperature range, the homogenizing temperature had little effect on the setting and final hardness values of butter. Thus the homogenizing temperature used in these experiments was maintained within this range.

Table 3 summarizes the results of twenty representative churnings, showing the percentage increase in hardness of butter from homogenized cream over that of butter from unhomogenized cream at three intervals following manufacture. The hardness increase resulting from homogenizing was usually most marked at five hours after manufacture, and decreased in magnitude when the butter was stored at $40^{o}F$ for seven days and thirty days.

Setting curves for butters from representative churnings of summer and winter creams are presented in Figure 4. In all cases, butter from homogenized cream set more rapidly than did butter from unhomogenized cream when both lots of cream received the same cooling treatment. The final hardness, measured after thirty days' storage at $40^{o}F$, was generally slightly higher for butters from homogenized cream at both seasons.

FIGURE 3 EFFECT OF CREAM HOMOGENIZING TEMPERATURE ON SETTING AND HARDNESS OF EXPERIMENTAL BUTTERS

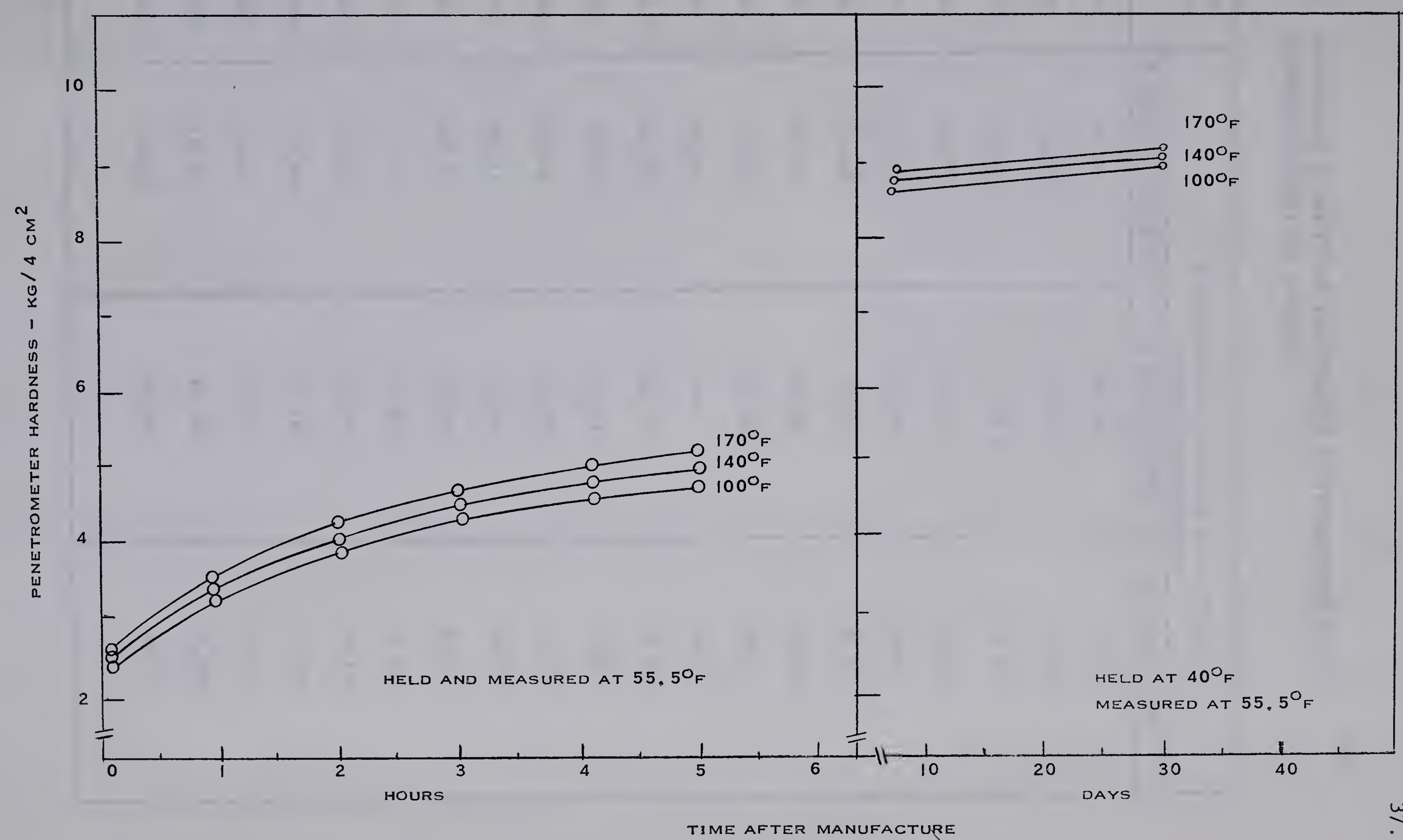
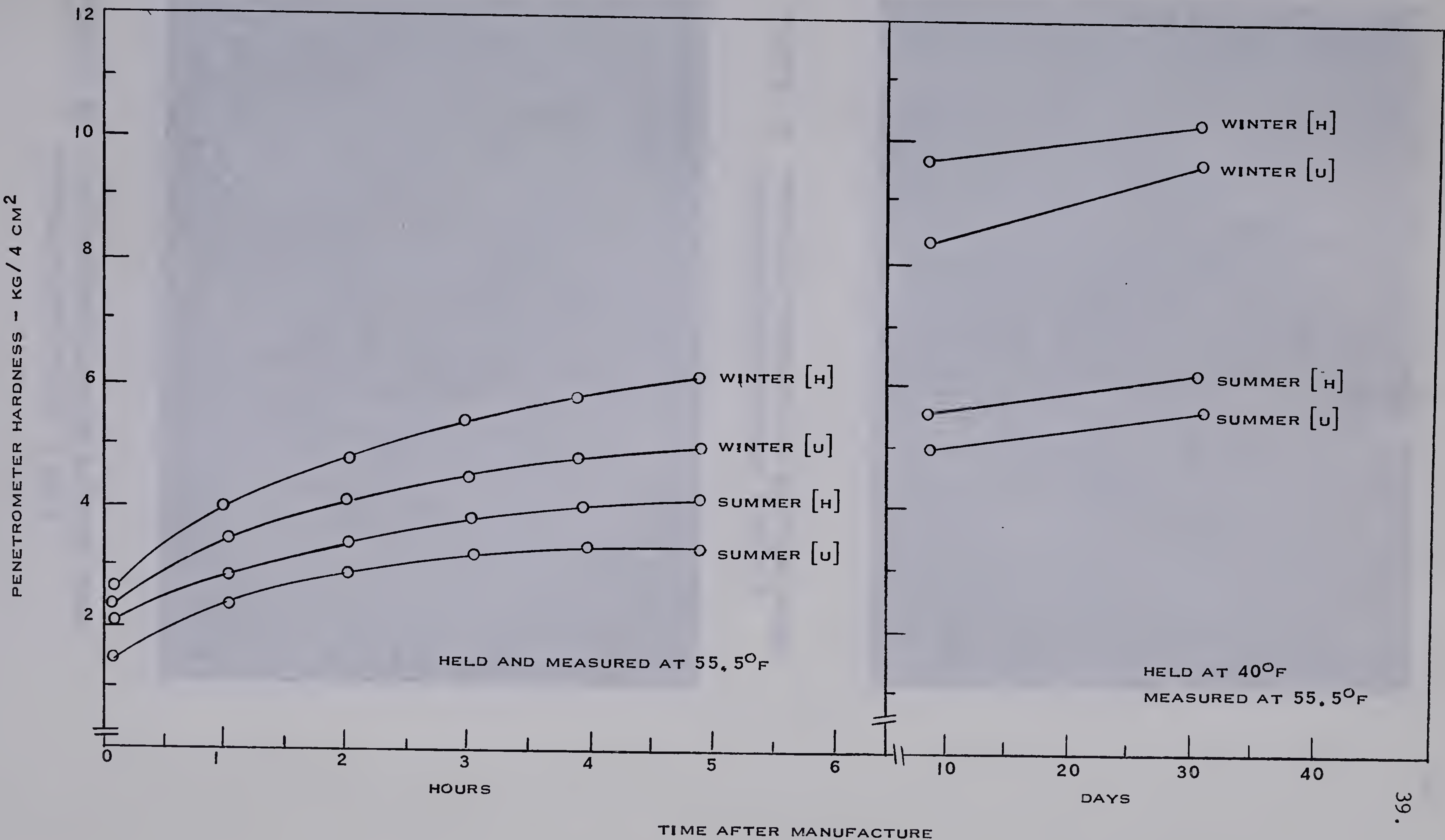


TABLE 3 INCREASE IN BUTTER HARDNESS ATTRIBUTABLE TO HOMOGENIZING OF CREAM

DATE OF CHURNING	% INCREASE IN HARDNESS AT INTERVALS AFTER MANUFACTURE		
	5 HOURS AT 55.5°F	7 DAYS AT 40°F	30 DAYS AT 40°F
4/6/65	42.81	39.48	31.25
17/6/65	14.28	13.96	7.80
23/6/65	12.34	7.38	5.95
29/6/65	19.77	6.93	6.38
5/7/65	7.14	7.54	6.18
17/8/65	3.92	5.78	0.00
17/8/65	38.42	17.50	16.99
18/8/65	58.97	27.68	26.31
3/9/65	29.41	——	4.54
8/10/65	23.71	16.77	9.50
14/10/65	40.00	9.33	1.56
15/10/65	19.55	8.08	4.39
3/11/65	28.12	11.62	20.13
5/11/65	29.27	13.56	8.83
18/11/65	7.77	4.93	1.57
19/11/65	22.54	14.70	3.84
3/12/65	22.90	11.55	7.57
8/12/65	31.21	11.03	13.15
10/12/65	13.22	10.90	0.00
11/12/65	22.22	10.90	8.61

FIGURE 4 SETTING AND HARDNESS OF REPRESENTATIVE SUMMER AND WINTER BUTTERS FROM HOMOGENIZED CREAM [H] AND UNHOMOGENIZED CREAM [U]



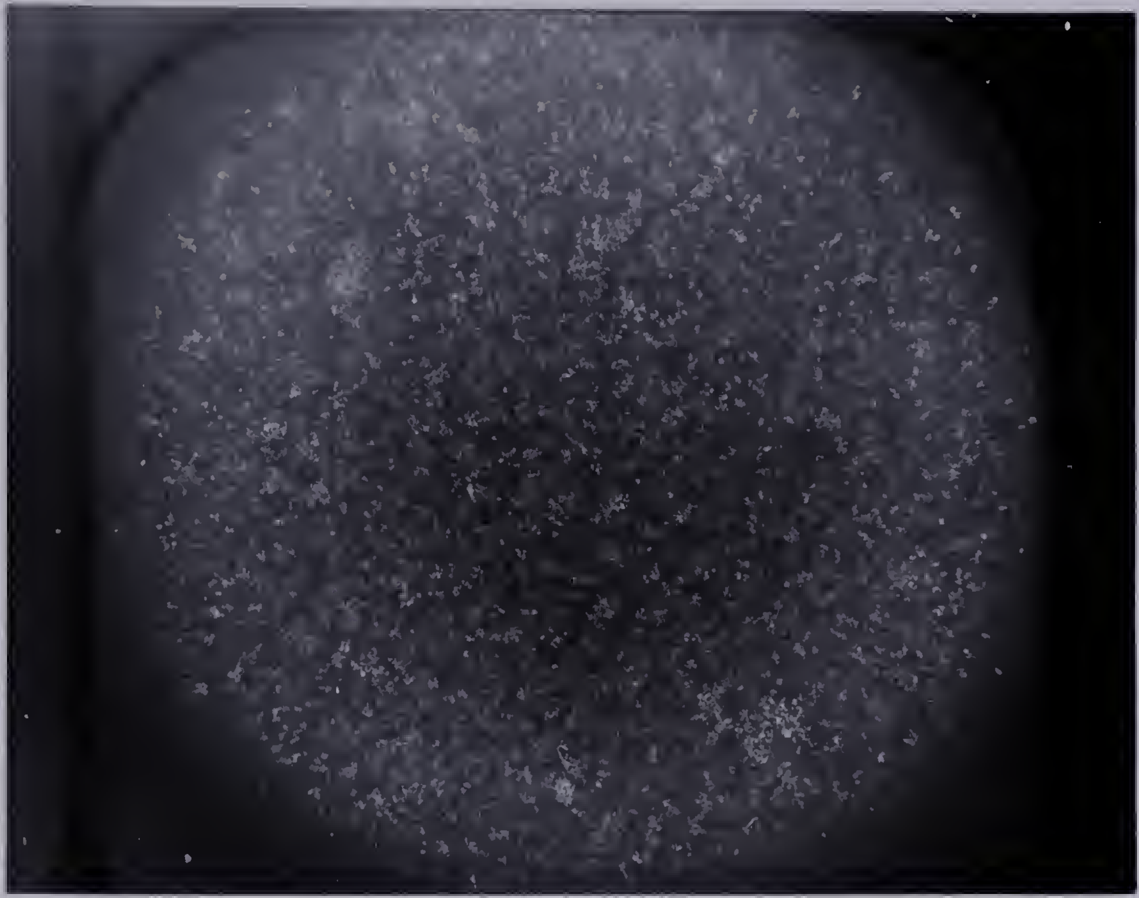


FIGURE 5A PHOTOMICROGRAPH OF BUTTER FROM UNHOMOGENIZED CREAM [500 X]

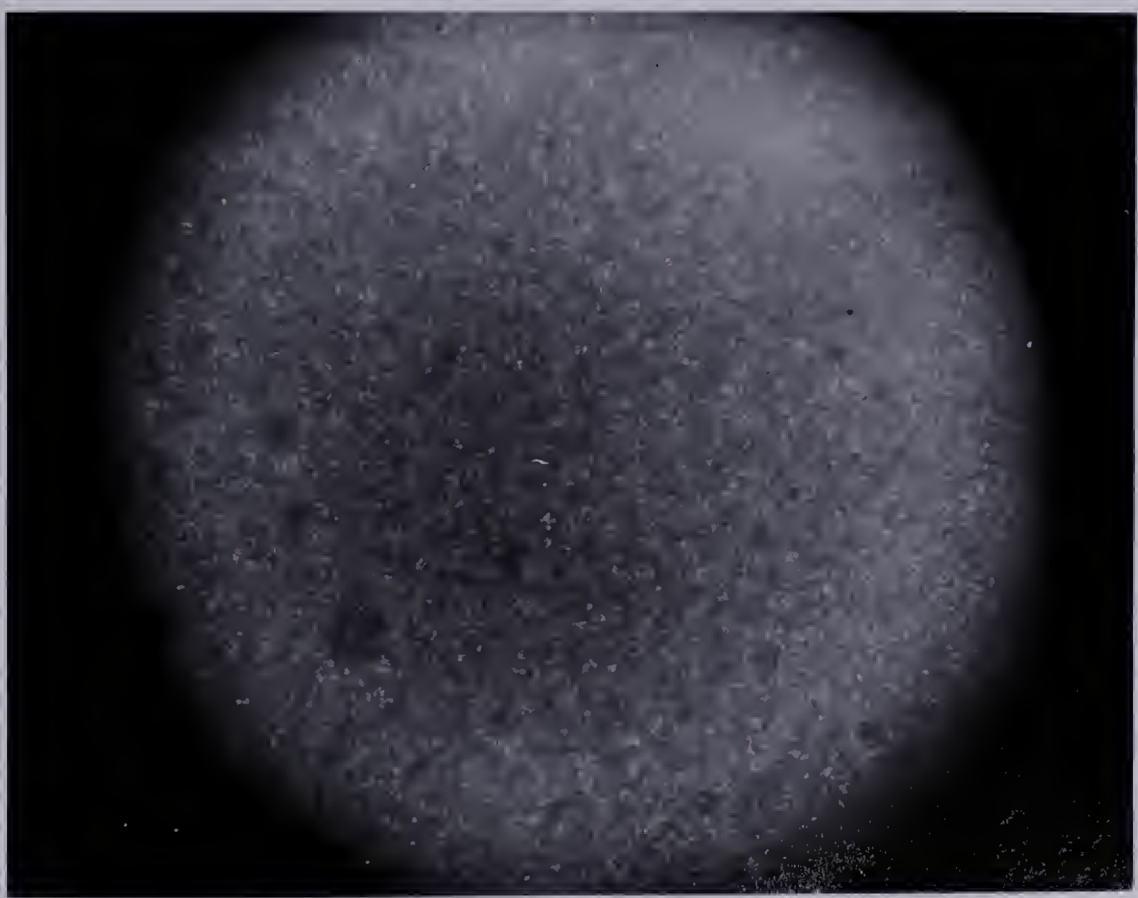


FIGURE 5B PHOTOMICROGRAPH OF BUTTER FROM HOMOGENIZED CREAM [500 X]

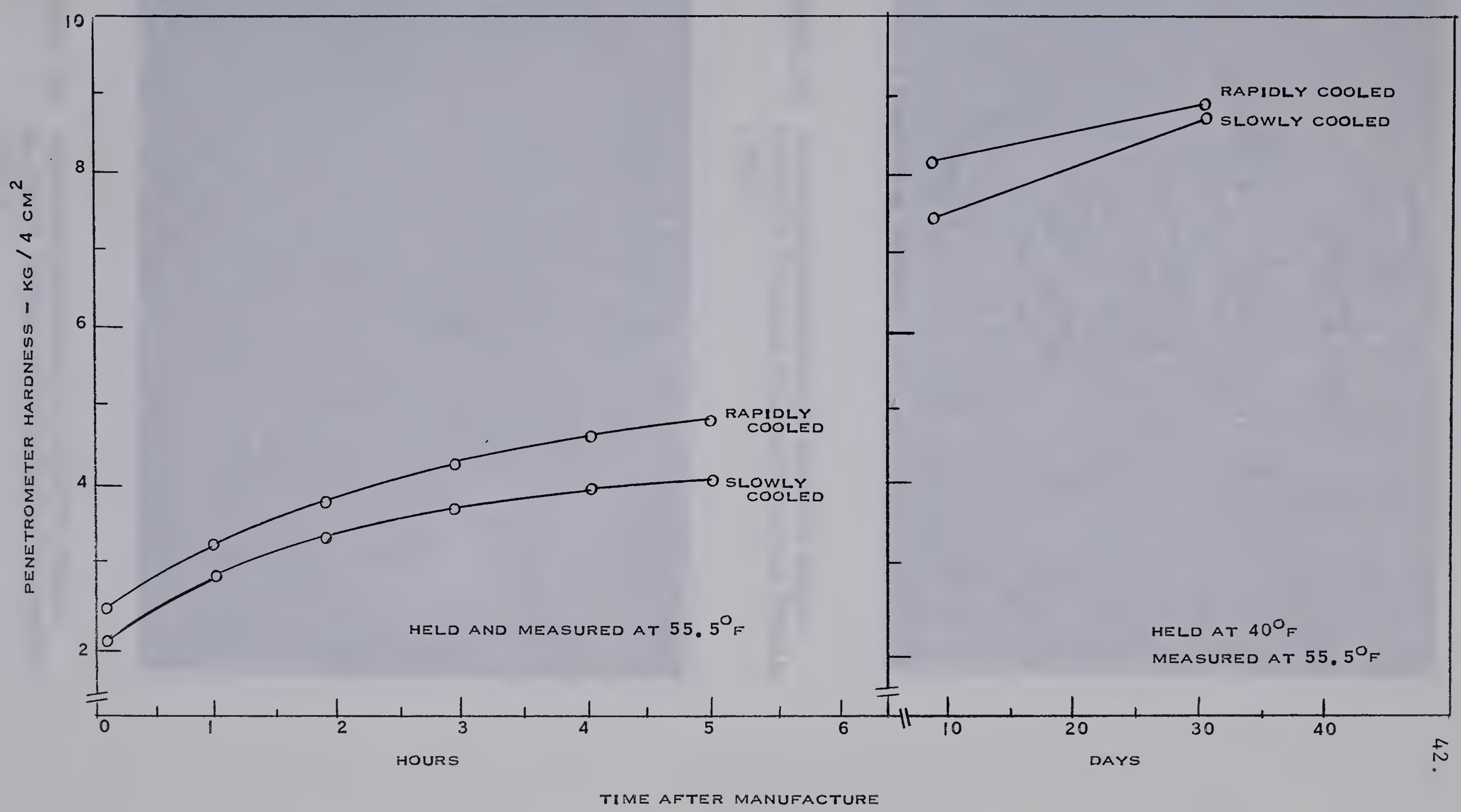
Figures 5A and 5B show the appearance of typical butters churned from unhomogenized and homogenized creams which were cooled identically. Homogenizing of cream in these experiments reduced the diameter of the majority of the fat globules in cream to less than 3 microns. The polarized light photomicrographs reveal that the butter from homogenized cream appears to contain a larger number of very small crystals than does butter from unhomogenized cream. Virtually no intact fat globules are visible in butter from homogenized cream; very few intact globules can be seen in butter from unhomogenized cream.

2. Rate of Cooling Cream

The effect of the cream cooling rate on setting and hardness values of representative butters from unhomogenized cream is shown in Figure 6. Higher setting rates and hardness values were obtained for butters from rapidly cooled cream. The final hardness values of butters from slowly cooled cream tended to approach those of butters from rapidly cooled cream. Polarized light photomicrographs (Figures 7A and 7B) indicate that there seems to be a greater amount of crystalline fat in the butter from rapidly cooled cream. Crystal aggregates are visible in both preparations.

The rate of cooling of homogenized cream apparently produced little or no change in butter setting or hardness values, as shown in Figure 8. Butter from slowly cooled homogenized cream generally exhibited setting and hardness values similar to those for butter from rapidly cooled cream. No differences were apparent when a number of these butters were examined under polarized light.

FIGURE 6 SETTING AND HARDNESS OF TYPICAL BUTTERS FROM RAPIDLY COOLED AND SLOWLY COOLED UNHOMOGENIZED CREAM



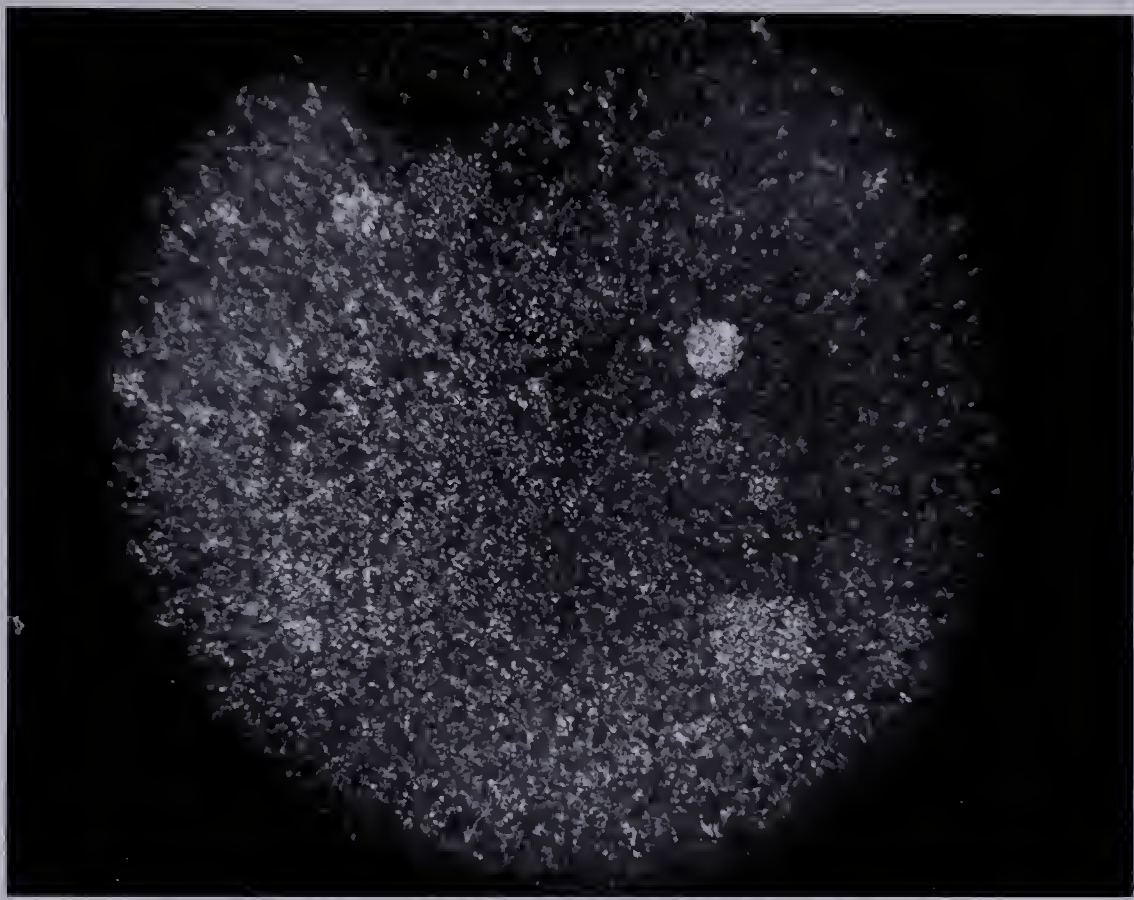


FIGURE 7A PHOTOMICROGRAPH OF BUTTER FROM RAPIDLY COOLED UNHOMOGENIZED CREAM [500 X]

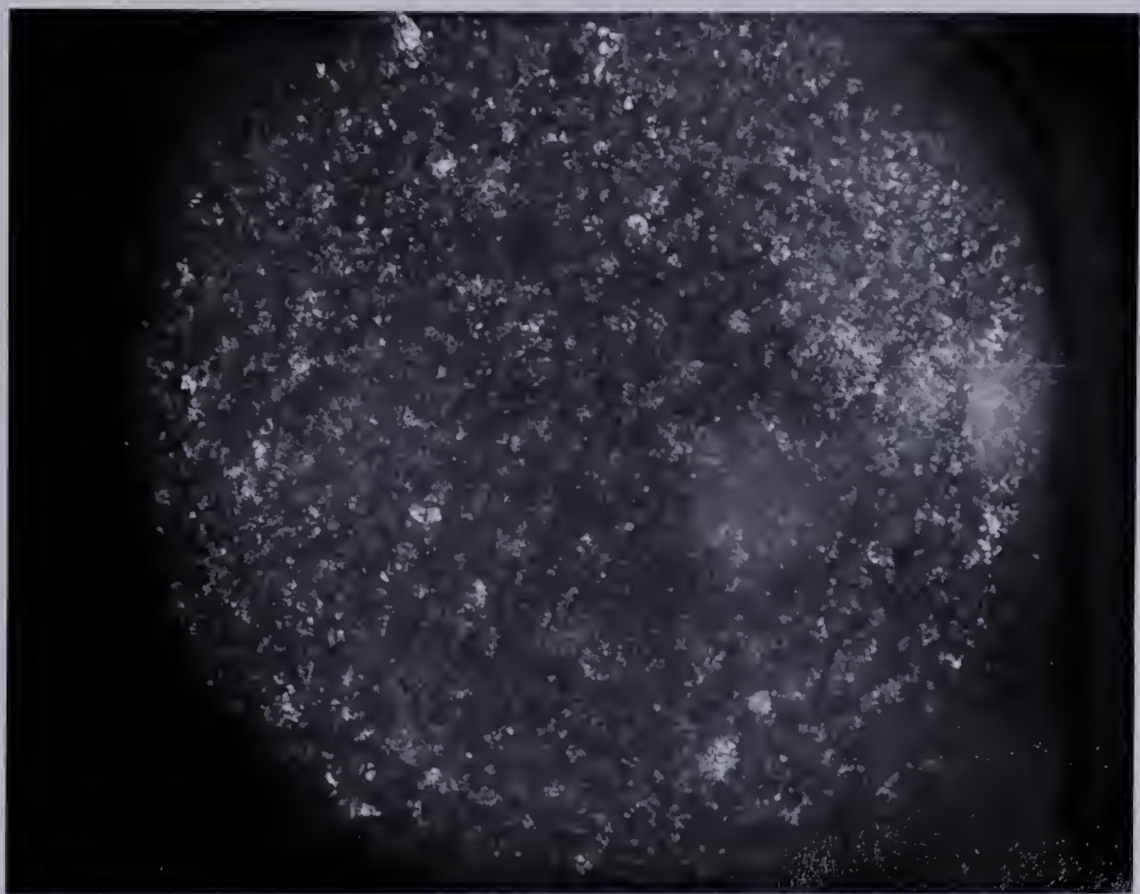
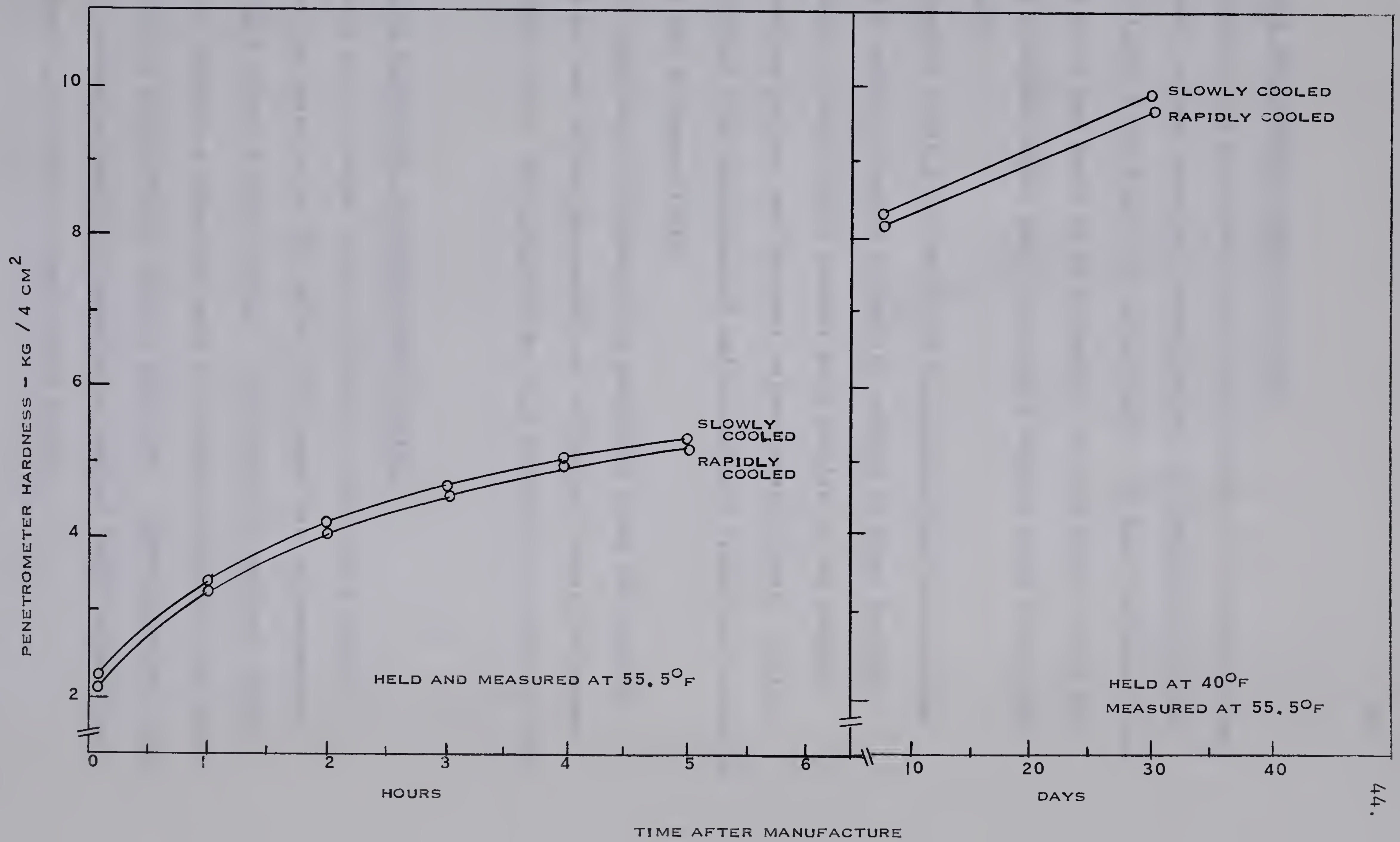


FIGURE 7B PHOTOMICROGRAPH OF BUTTER FROM SLOWLY COOLED UNHOMOGENIZED CREAM [500 X]

FIGURE 8 SETTING AND HARDNESS OF TYPICAL BUTTERS FROM RAPIDLY COOLED AND SLOWLY COOLED HOMOGENIZED CREAM



3. Precooling and Stepwise Cooling of Cream

Butters from precooled cream (40-65-50) set less markedly than did the control butters from both unhomogenized and homogenized creams, as shown in Figure 9 and Figure 10 respectively. In both instances, the hardness reduction was found to be permanent, in that butters from precooled cream remained softer than the control butters after thirty days' storage at 40°F.

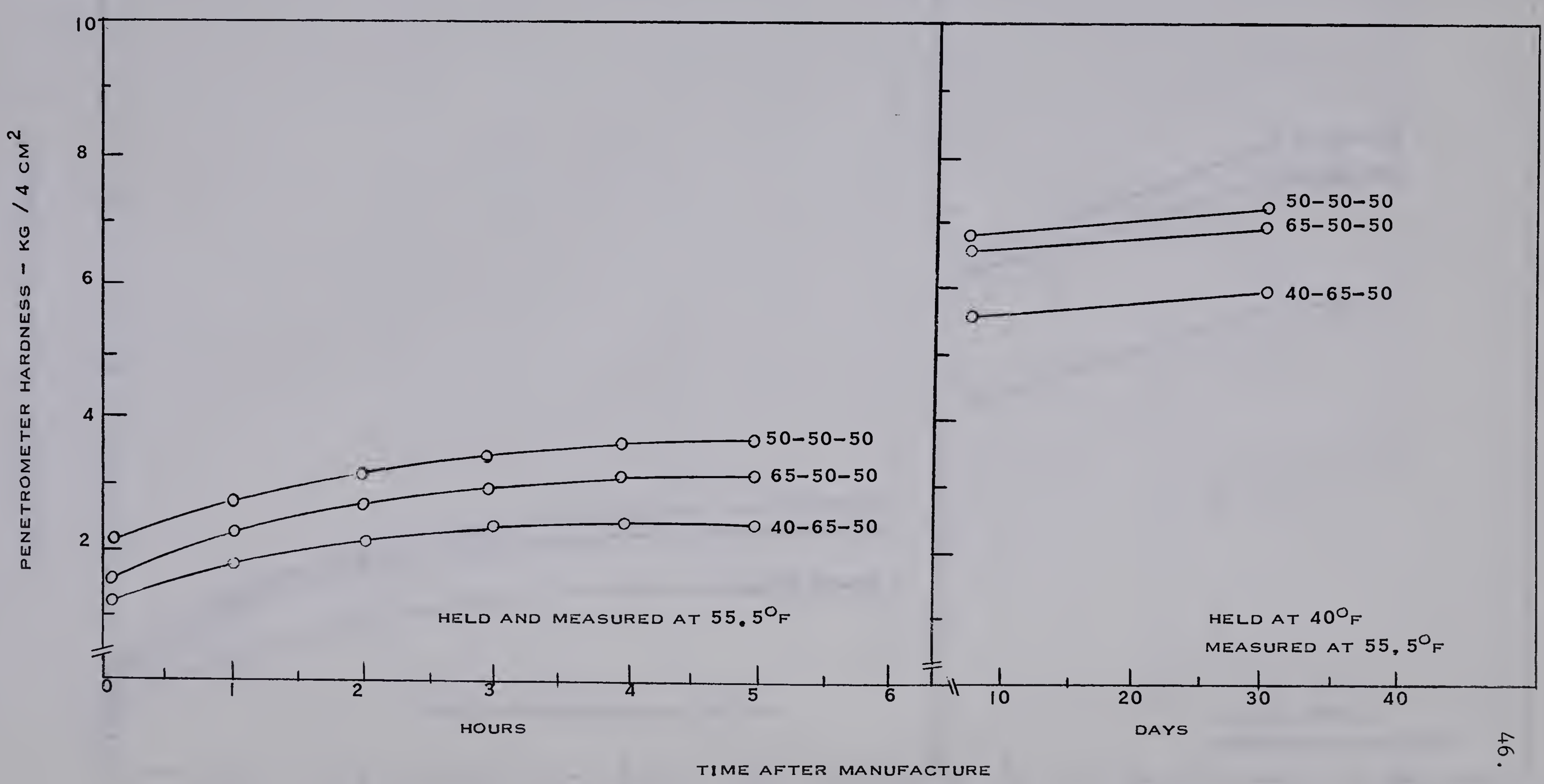
Stepwise cooling (65-50-50) of homogenized and unhomogenized creams did not appear to reduce either the setting or final hardness values of butter. The 65-50-50 butters were similar to the control butters in setting pattern and hardness values in all trials. Typical curves for butters from unhomogenized and homogenized creams are presented in Figures 9 and 10 respectively.

The appearance of butters from precooled cream and stepwise cooled cream did not differ noticeably nor was either visibly different from the control butter when preparations were examined by polarized light microscopy.

4. Addition of Liquid Fat to Cream before Cooling

There was no change in the setting of butter which could be attributed to the addition of 5%, 10% or 15% liquid fat to homogenized or unhomogenized creams before cooling. The thirty-day hardness values of the butters containing added fat were not notably different from those of control butters which did not contain added fat. Under polarized light, some butters containing added fat appeared to contain large, bright aggregates which were not visible in the control butter.

FIGURE 9 EFFECTS OF PRECOOLING AND OF STEPWISE COOLING OF UNHOMOGENIZED CREAM ON SETTING AND HARDNESS OF TYPICAL EXPERIMENTAL BUTTERS



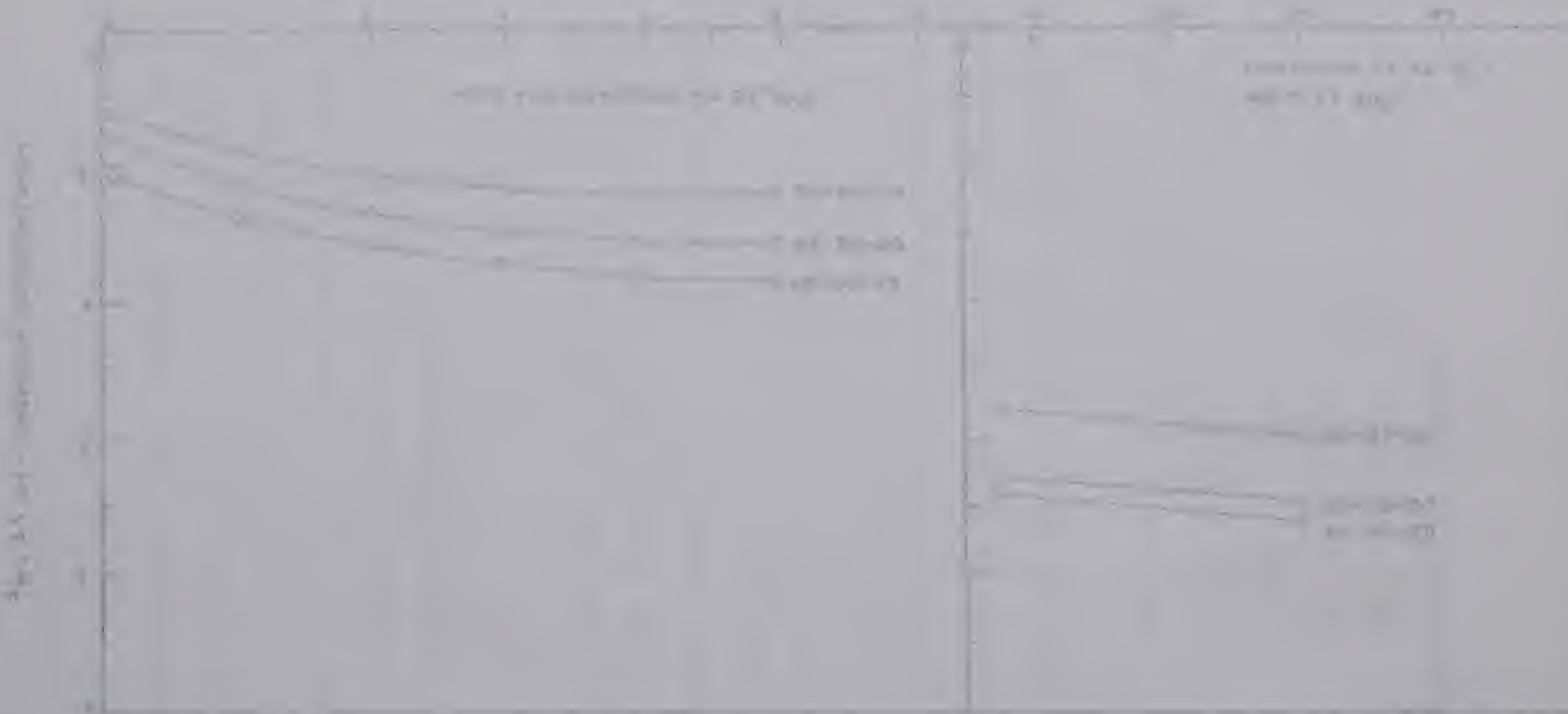
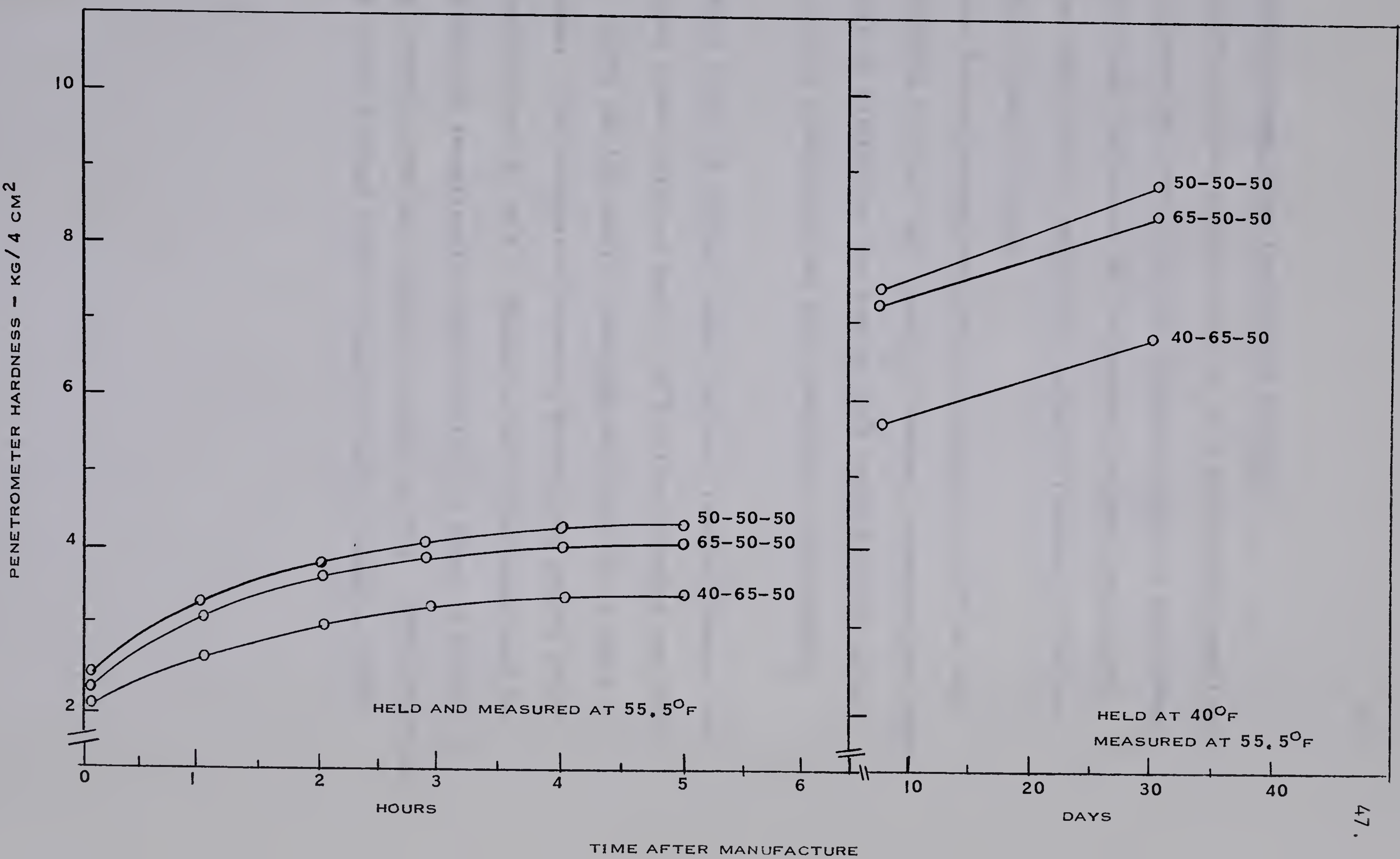


FIGURE 10 EFFECTS OF PRECOOLING AND OF STEPWISE COOLING OF HOMOGENIZED CREAM ON SETTING AND HARDNESS OF TYPICAL EXPERIMENTAL BUTTERS



5. <u>Time Interval between Cream Cooling and Churning</u>

Homogenized cream held one hour at 40°F and churned immediately yielded butter which set rapidly to high hardness values in both summer and winter churnings. Extension of the interval between cooling and churning to two hours did not produce any great differences in the setting or hardness values of butter.

Butter from unhomogenized winter cream held at 40°F for one hour before churning also set very rapidly. However, unhomogenized summer cream churned after holding for one hour at 40°F yielded softer butter than did the control cream which was held overnight at the churning temperature.

Setting curves of typical summer butters from both homogenized and unhomogenized creams held one hour at 40°F before churning are shown in Figure 11. Cream for the control churnings was held for 18 hours at 45°F in the summer. The hardness differences became less marked after storage of summer butters at 40°F. Winter butters from cream held one hour at 40°F exhibited exceptionally rapid setting, as shown in Figure 12. In winter, the control cream was held for 18 hours at 50°F. The difference in hardness of winter butters tended to persist after storage at 40°F.

All butters from cream held one hour and churned immediately were crumbly and brittle whereas butters from cream held overnight were smooth and even in texture. In spite of this difference in outward appearance, the control butters and butters from short-hold churnings did not appear different when examined under polarized light.

FIGURE II INFLUENCE OF TIME INTERVAL BETWEEN COOLING AND CHURNING OF CREAM ON SETTING AND HARDNESS OF SUMMER BUTTERS FROM HOMOGENIZED CREAM [H] AND UNHOMOGENIZED CREAM [U]

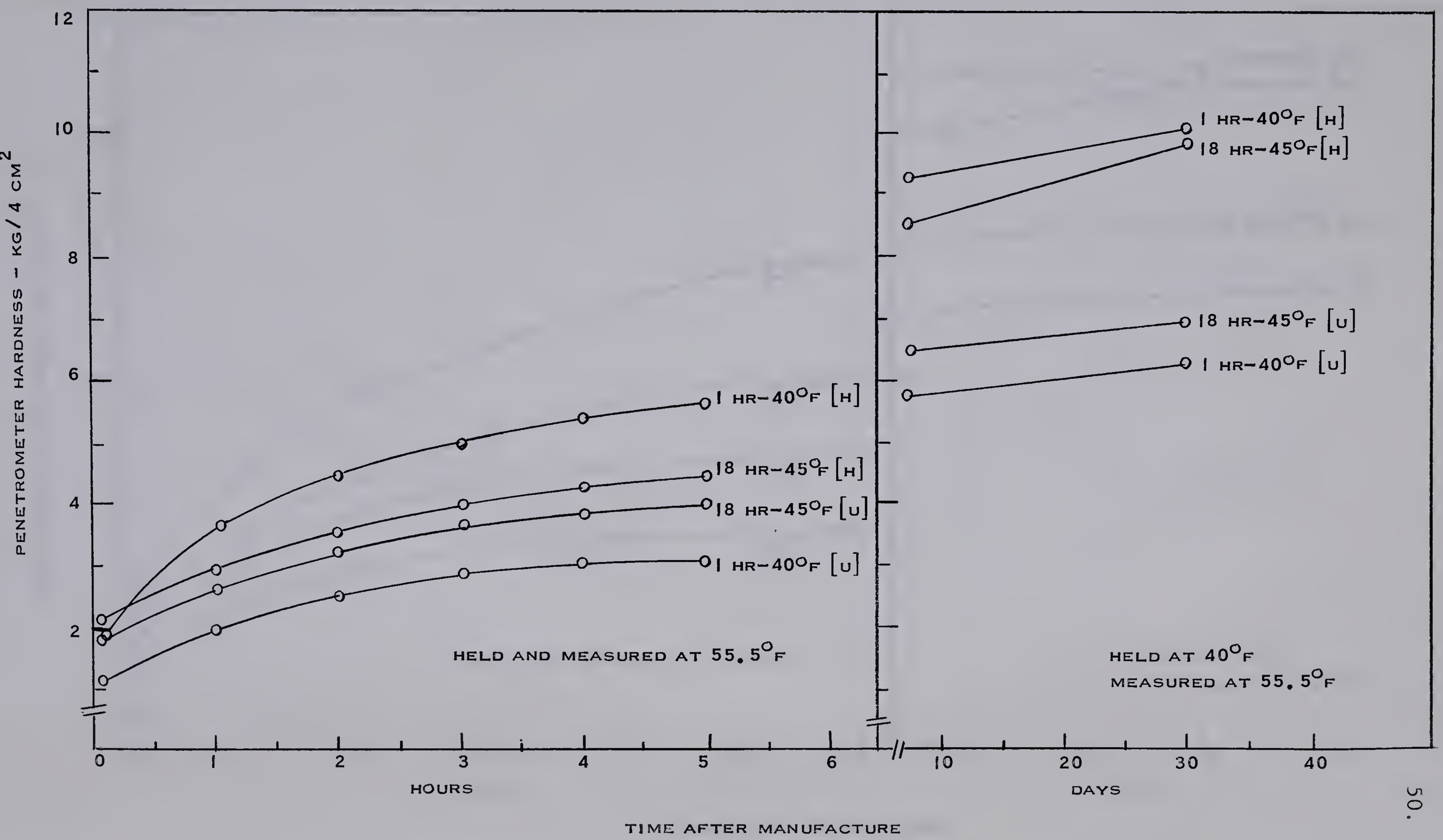
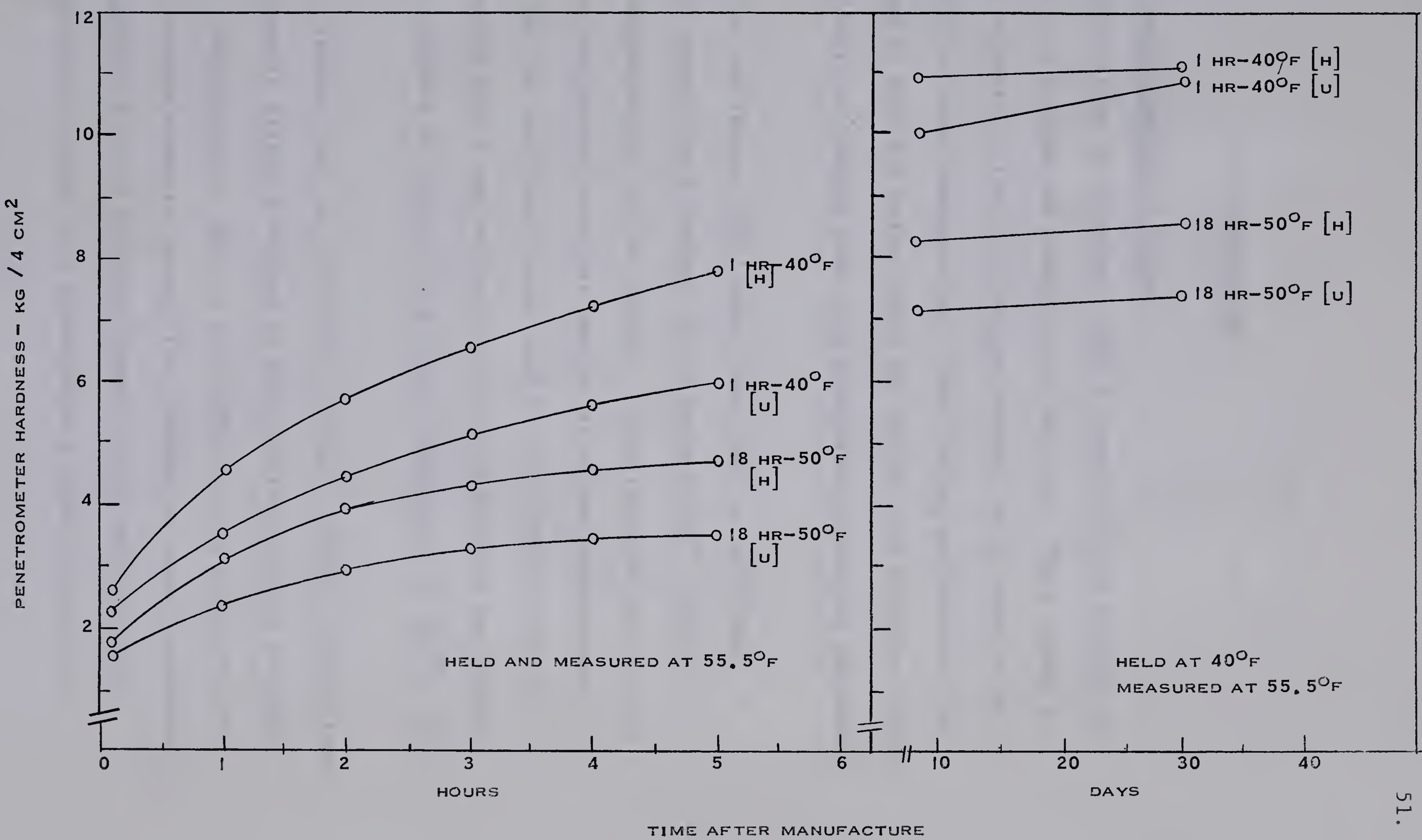


FIGURE 12 INFLUENCE OF TIME INTERVAL BETWEEN COOLING AND CHURNING OF CREAM ON SETTING AND HARDNESS OF WINTER BUTTERS FROM HOMOGENIZED CREAM [H] AND UNHOMOGENIZED CREAM [U]



V. DISCUSSION

1. Effect of Homogenization of Cream

Homogenization of cream was found to promote rapid setting of butter held at 55.5°F after the completion of working. There seemed to be very little increase in the final butter hardness which could be attributed directly to cream homogenizing. This finding corroborates the results obtained by Wood and Dolby (1965) for butter from Vacreated cream and by Dolby (1965) for butters made from homogenized cream and Vacreated cream.

Goulden and Phipps (1964) have shown that the efficiency of homogenizing is temperature dependent in the range 40-80°C (104-176°F) for creams of approximately 30% fat content. They reported that the fat globule size in homogenized cream was smaller at higher temperatures. Nevertheless, for the temperature range used in the present study, no obvious temperature effect was evident, as little difference was noted in the setting or hardness of butters from creams homogenized at 100°F, 140°F or 170°F.

It is assumed that the increased setting of butters churned from homogenized creams is directly related to the size reduction of the milk fat globules. Homogenizing at 800 psi was found to cause a distinct reduction in the average diameter of milk fat globules in cream; consequently, butter from homogenized cream contained numerous very small crystals (Figure 5B) compared to butter from unhomogenized cream (Figure 5A).

The actual amount of solid fat in butter from homogenized cream might not differ greatly from that in butter made from unhomogenized cream cooled identically, so that the final hardness of both butters would be similar. However, the setting rate of the butter from homogenized cream would be higher because of the increased quantity of thixotropic crystals present.

It was also noted that almost no intact fat globules were visible in butters churned from homogenized cream. Similarly, Wood and Dolby (1965) reported that intact milk fat globules could not be seen in butter made from Vacreated cream. It is possible that some of the extremely small globules might not be visible with the magnification used in polarized light microscopy, or that the vigorous agitation during churning in the modified freezer caused a greater-than-usual breakup of the fat globules in cream. A reduction in the number of intact fat globules in butter would also indicate that a greater number of small crystals had been released into the free fat phase of the butter during churning and working; thus the likelihood of thixotropic setting would be considerably increased.

Some relationship apparently exists between the effects of Vacreation and those of homogenization of cream, since both treatments tend to increase the number of small fat globules in cream and, thereby, also increase the quantity of small crystals in butter. Both the decreased crystal size and larger proportion of thixotropic particles would tend to promote butter setting.

2. Influence of the Cream Cooling Rate

Rapid and slow cooling of unhomogenized cream produced the anticipated effects on butter setting and hardness. These findings confirm those of Huebner and Thomsen (1957b) and de Man and Wood (1959a) that slow cooling of cream resulted in softer butter. The aggregates which can be seen in the photomicrographs (Figures 7A and 7B) appear to be groups of crystals, similar to those observed by de Man (1964a) in tempered milk fat. These aggregates do not seem to affect the hardness or setting of the butters.

The rate of cooling of homogenized cream did not have any marked influence on the settings or hardness values of butters (Figure 8). Dolby (1954) stated that butter from slowly-cooled Vacreated cream was softer than butter from rapidly cooled cream. It thus appears that, unlike Vacreation, homogenization obviates the usual effect of the cream cooling rate on the setting and hardness of butter. Neither the quantity nor the size of crystals in butters from homogenized cream seemed to be affected by the cream cooling rate, as the butter preparations were similar in appearance under polarized light. On the basis of these observations, it might be assumed that the solid fat content of the homogenized cream was not reduced by the slow cooling procedure used in this study or that any decrease in the solid fat content was insufficient to produce a measurable change in setting or hardness of butter. The mode of milk fat crystallization in the finely dispersed globules of homogenized cream may also have been altered in such a way that the physical properties of the butter were changed.

3. Effects of Precooling and Stepwise Cooling of Cream

a. Precooling

Butters churned from precooled homogenized and unhomogenized creams exhibited consistently lower rates of setting and lower final hardness values than did butters from either the control cream or stepwise-cooled cream. The findings for butters made from unhomogenized cream confirmed those reported by Wood and Dolby (1965) for butter from plate-pasteurized cream. However, the results for butters made from homogenized cream did not substantiate the findings of Dolby (1959) for butters from precooled Vacreated cream. Dolby found that precooling of Vacreated cream was no more effective than was stepwise cooling in lowering butter hardness. Further work by Wood and Dolby (1965) confirmed that Vacreation of cream impaired the effectiveness of precooling in reducing butter hardness. Evidently homogenization does not inhibit the additional softening effect of cream precooling; thus Vacreator treatment and homogenizing may differ in some essential aspect which might account for the variation in the effects of precooling noted in this study.

Control butters and butters from precooled cream appeared similar in crystal structure under polarized light, though the hardness of the latter butter was lower. According to de Man (1964a), tempering of milk fat generally reduced its hardness but the crystal size was not much different from that in rapidly cooled milk fat. This observation may apply to the butters observed in the present study as well as to milk fat.

b. Stepwise Cooling

Churning of stepwise-cooled homogenized cream produced butter

which set as quickly as the control butter and attained the same final hardness. Butters from stepwise-cooled cream were also found to be harder at all stages than were butters made from precooled cream. By contrast, Dolby (1959) reported that stepwise-cooled Vacreated cream yielded butter which was considerably softer than the control butter from rapidly cooled cream, but was similar in hardness to butter churned from precooled cream.

Although stepwise cooling of cream would be expected to lower its solid fat content and result in softer butter, as observed by Dolby (1959), no lowering of the setting rate or final hardness values occurred for butter from stepwise-cooled homogenized cream in the present study. This finding seems to indicate further that homogenizing and Vacreation of cream are essentially different in their influence on butter hardness and setting under certain conditions. The cause of this difference has not been determined. It is possible that the crystallization of milk fat during stepwise cooling of cream is changed by Vacreation but is not affected by homogenization.

Butters made from stepwise-cooled unhomogenized cream were also similar to the control butters in setting and hardness values; in addition, they were always harder than butters churned from precooled cream. These results substantiate the findings of Wood and Dolby (1965) for butter from stepwise-cooled plate-pasteurized cream, which set more rapidly than did butter from precooled cream and remained harder after 40 days' storage.

The similarities in hardness values and setting patterns found for control butters and butters from stepwise-cooled cream may be ex-

plained on the basis of an investigation of cooling treatments applied to milk fat (de Man, 1964a). Although the solid fat content of the butters made from stepwise-cooled cream may actually be lowered, the crystal structure might also be changed so that it imparts additional hardness to the butter; thus the solid fat content may not be the most important factor involved in this treatment.

There were no obvious differences in the size or quantity of crystals in preparations of butters made from stepwise-cooled cream or control butters which were examined under polarized light. Differences in crystal form might have occurred but would not be readily observable in the butter preparations, though they were clearly shown in photomicrographs of pure milk fat presented by de Man (1964a).

4. Addition of Liquid Butterfat to Cream

Neither the setting nor the final hardness of butters from homogenized or unhomogenized creams was influenced by the addition of small quantities of liquid fat to the cream before cooling. The bright aggregates which could be seen in preparations of the butters observed under polarized light may have been caused by the coalescence of the extra fat to form the large particles visible in the butter. These aggregates would be too large to contribute to thixotropic setting and yet did not appear to be sufficiently numerous to effect any measurable hardness increase in the butter.

Dolby (1953) noted that a few large aggregates of fat were present in butter from Vacreated cream observed under polarized light. However, these particles were not considered to be a primary factor in-

fluencing the physical properties of the butter. It is possible, therefore, that such fat masses are not of great importance in determining the setting or hardness of butters made from either Vacreated cream or homogenized cream.

5. Influence of the Time Interval between Cooling of Cream and Churning

Butters from summer and winter homogenized creams which were cooled rapidly to 40°F and held one or two hours before churning exhibited unusually rapid setting during the five hours after manufacture. A similar phenomenon was observed by Wood and Dolby (1965) for butters from Vacreated cream. In both instances, the final hardness of these butters was not greatly different from that of the control butters.

According to the generally accepted view, cream held overnight at the churning temperature contains a maximum amount of solid fat and should thus yield the hardest possible butter at any season. Similarly, butter churned from cream held for only one or two hours might be expected to be soft because crystallization of the milk fat in cream would certainly be incomplete at the time of churning. However, crystal formation would be initiated very promptly in cream cooled to 40°F. In addition, agitation during churning could further promote crystallization by increasing nucleation. It is possible that crystal formation could proceed in these butters during working and holding for 5 hours at 55.5°F. Such continuing crystallization might be compared to that reported by de Man and Wood (1958a) for continuously-made butter.

Polymorphic transformations have been suggested as a possible cause of the rapid setting in butters churned from cream held at 40°F

for one hour (Wood and Dolby, 1965). Although polymorphism is not usually regarded as a likely cause of setting in conventionally-made butter, it could be a factor in this particular instance. The effects of the short interval between cream cooling and churning were most marked during the first 5 hours after working; it has been suggested by de Man and Wood (1959b) that polymorphism would tend to have a short term effect on the properties of butterfat and butter.

In the present study, butters from unhomogenized creams held for one hour before churning also exhibited unusually rapid setting in winter; however, summer butters set less markedly than the control butter and the final hardness was lower. This discrepancy may be caused by the glyceride composition of the milk fat in summer cream. A limited number of crystal centres would probably be formed on cooling of summer cream; thus the low setting rate and low final hardness of the butter might be expected.

The butters from 'short-hold' churnings and the control butters were quite different in outward appearance, especially in cross-section. The very coarse texture of the 'short-hold' butters suggests that large crystals might be responsible for their appearance. Nevertheless, there were no readily discernible differences in crystal size or number of crystals seen in preparations of the butters which were examined under polarized light. If large crystals or crystal aggregates were present, they may have been pressed out by the cover slip when the slide was prepared for examination.

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VII. APPENDIX A

COMPARISON OF THREE METHODS OF MEASURING BUTTER HARDNESS

There are a number of instrumental procedures which can be used to measure butter hardness. It is often difficult to relate the results obtained by different methods. The three methods used in this investigation differ mainly in the mode of deforming the sample and in sample size. The measuring temperature and rate of shear were constant for all determinations.

From the analysis of the results, it is possible to ascertain the type of relationship that exists among the methods and to determine whether the same property of butter can be measured by any one of the methods.

1. Procedure for Measuring Butter Hardness

Penetration, sectility and extrusion were used to determine the hardness of sixty conventional butters and twenty continuous butters. The butters had set completely before testing. Duplicate samples for the three methods were cut from each butter. Round frames were used for penetration samples and slotted frames for sectility samples; a small block of butter was cut for extrusion samples. All samples were tempered at 55.5^{o}F for 24 hours before testing.

The apparatus used was an NIRD extruder with modifications for penetration and sectility measurements (Figure 2). A shear rate of 1.56 inch/minute was used throughout. All measurements were made in a room maintained at 40^{o}F.

The accuracy of each method was determined for both conventional and continuous butters from summer and winter churnings. The results are presented in Table A-1, indicating satisfactory accuracy in all cases.

Penetration results are expressed as kg/4 cm^2. The maximum force required to drive the disc to a depth of 1 cm into a butter sample held firmly in a stainless steel frame is recorded on a moving chart.

Sectility values are measured as the maximum force in kilograms required for a wire 0.046 cm in diameter and 5.0 cm long to cut through 1.9 cm thickness of a butter sample which is held in a slotted frame. Sectility values are expressed as kg/5 cm, since 5.0 cm is the length of the wire which actually contacts the butter surface during cutting.

Extruder hardness of butter samples was determined by the procedure outlined by Prentice (1954). Extrusion values are expressed in kilograms.

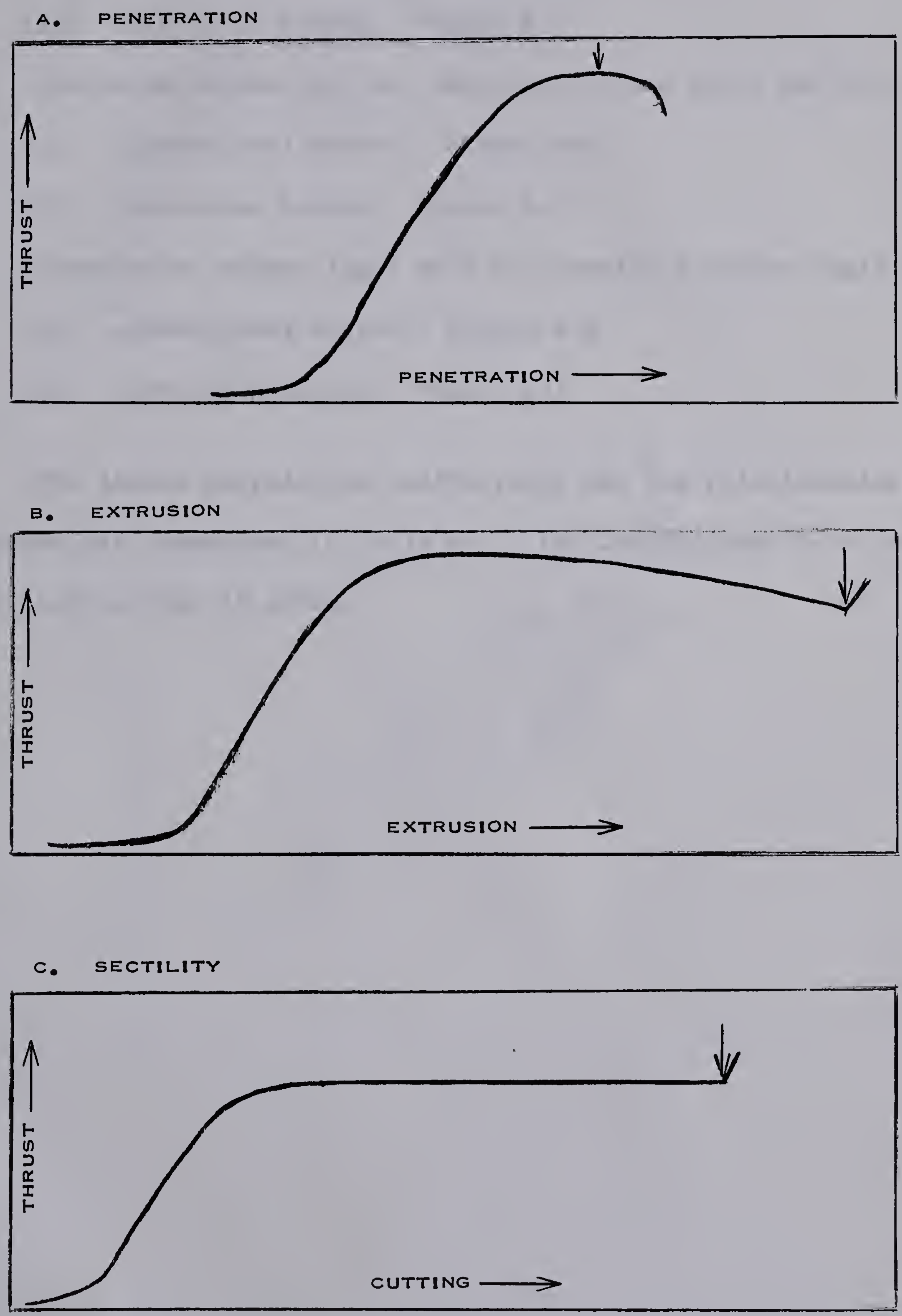
Typical traces for each method are illustrated in Figure A-1. The arrow above each trace indicates the point at which the reading is taken.

TABLE A-1 ACCURACY OF EXTRUSION, PENETRATION AND SECTILITY MEASUREMENTS

CONVENTIONAL BUTTER	PENETROMETER KG/4 CM^2		EXTRUDER KG		SECTILITY KG/5 CM	
	SUMMER	WINTER	SUMMER	WINTER	SUMMER	WINTER
RANGE - 10 SAMPLES	5.20-6.00	5.60-6.00	2.10-2.20	2.40-2.60	0.375-0.400	0.375-0.425
MEAN	5.68	5.81	2.17	2.51	0.390	0.390
AVERAGE DEVIATION FROM MEAN	±4.50%	±1.89%	±1.90%	±2.86%	±3.00%	±3.81%

CONTINUOUS BUTTER	PENETROMETER KG/4 CM^2		EXTRUDER KG		SECTILITY KG/5 CM	
	SUMMER	WINTER	SUMMER	WINTER	SUMMER	WINTER
RANGE - 10 SAMPLES	7.90-8.20	10.00-10.80	1.40-1.70	2.90-3.10	0.500-0.550	0.750-0.825
MEAN	8.04	10.46	1.50	3.01	0.545	0.800
AVERAGE DEVIATION FROM MEAN	±1.02%	±2.60%	±5.33%	±2.39%	±1.65%	±2.50%

FIGURE A-I TYPICAL TRACES OF PENETRATION, SECTILITY AND EXTRUSION DETERMINATIONS



2. Relationship of the Three Methods of Measuring Butter Hardness

Scatter diagrams were drawn for the following comparisons:

a. Penetration values (kg/4 cm^2) vs. extrusion values (kg) for-

(i) conventional butter: Figure A-2

(ii) continuous butter: Figure A-3

b. Extrusion values (kg) vs. sectility values (kg/5 cm) for-

(i) conventional butter: Figure A-4

(ii) continuous butter: Figure A-5

c. Penetration values (kg/4 cm^2) vs. sectility values (kg/5 cm) for-

(i) conventional butter: Figure A-6

(ii) continuous butter: Figure A-7

The linear correlation coefficients for the relationships described above are summarized in Table A-2. The coefficients were found to be significant at the 1% level.

FIGURE A-2 RELATIONSHIP BETWEEN PENETRATION AND EXTRUSION VALUES FOR 60 SAMPLES OF CONVENTIONAL BUTTER

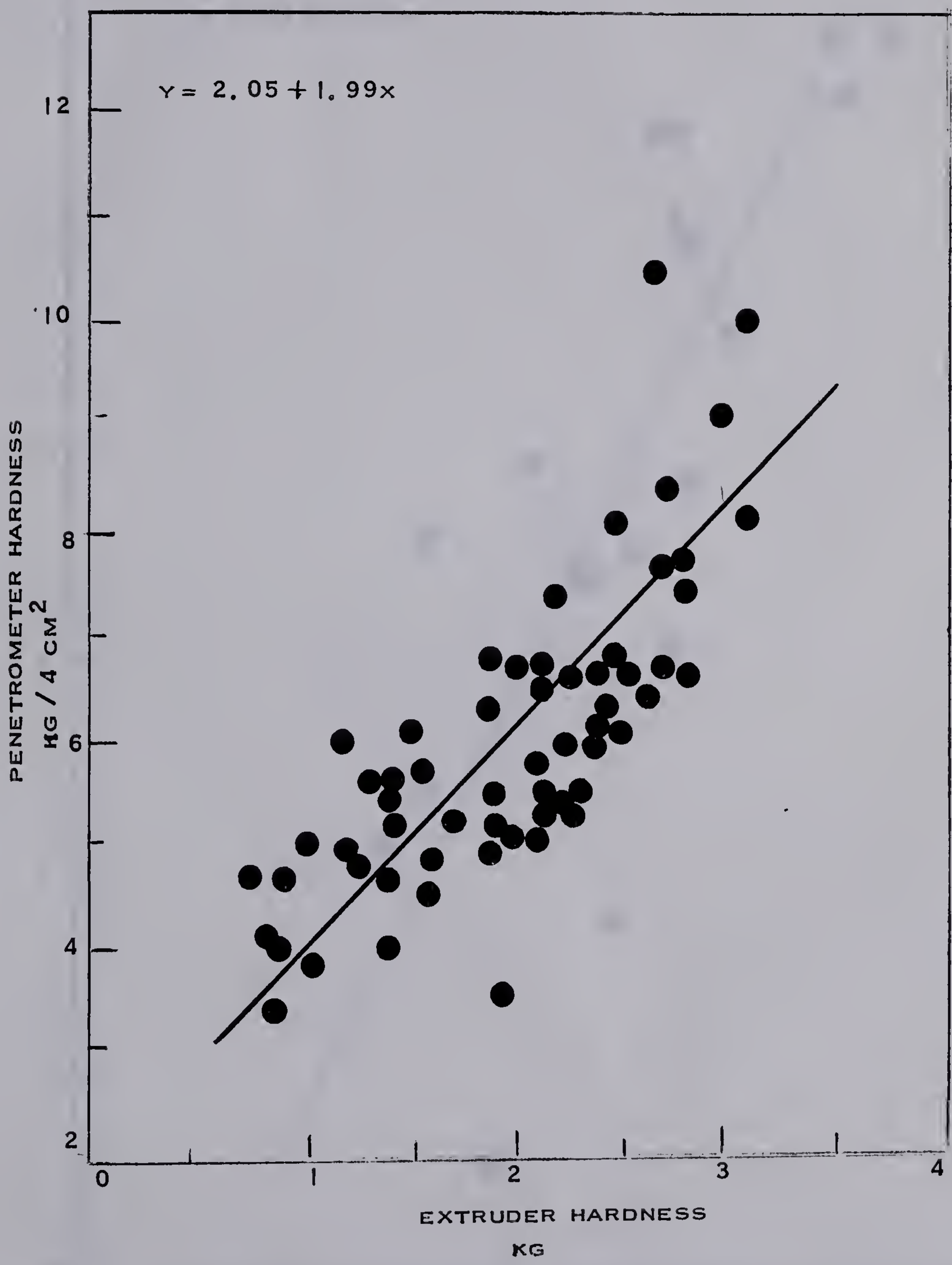


FIGURE A-3 RELATIONSHIP BETWEEN PENETRATION AND EXTRUSION VALUES FOR 20 SAMPLES OF CONTINUOUS BUTTER

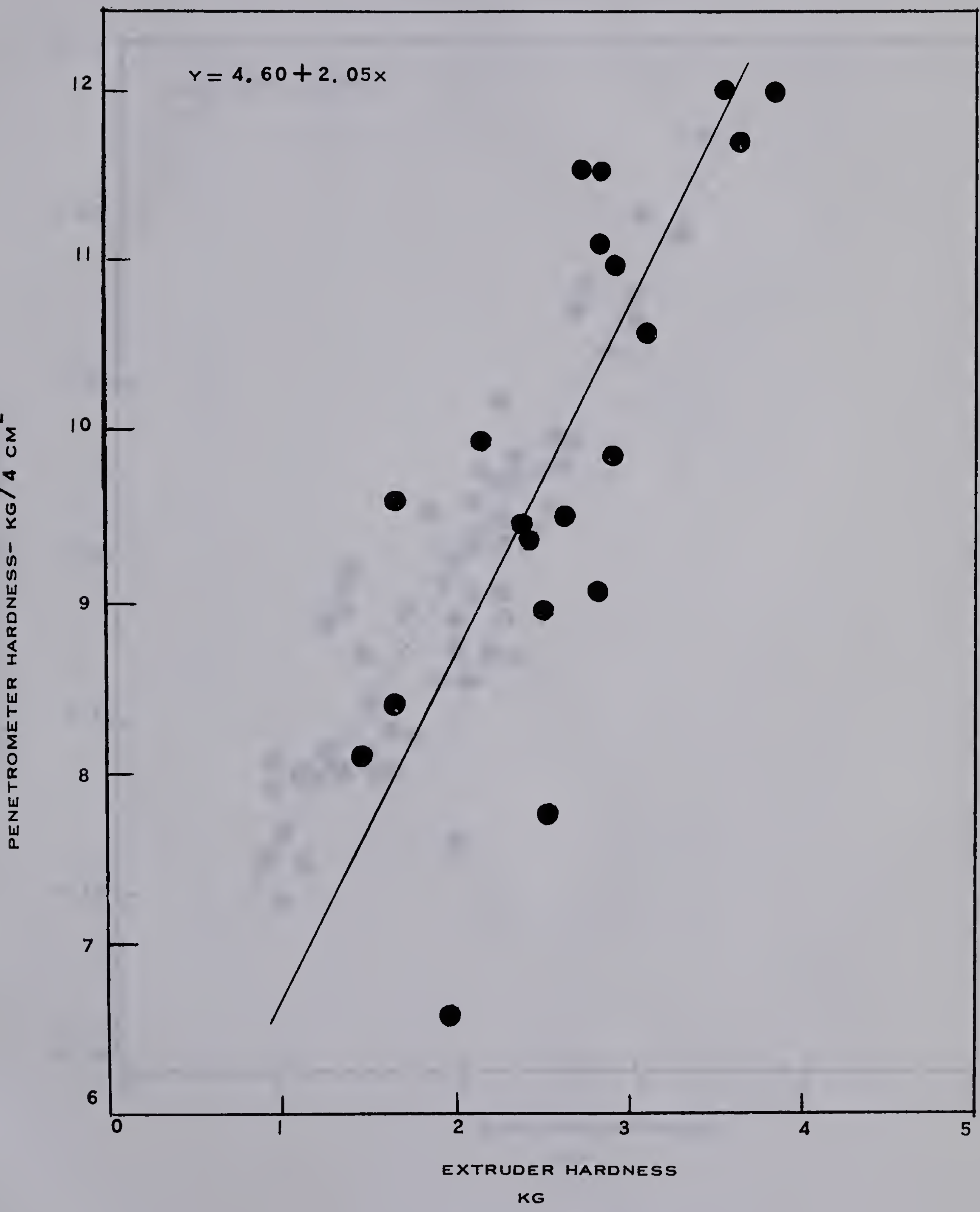


FIGURE A-4 RELATIONSHIP BETWEEN EXTRUSION AND SECTILITY VALUES FOR 60 SAMPLES OF CONVENTIONAL BUTTER

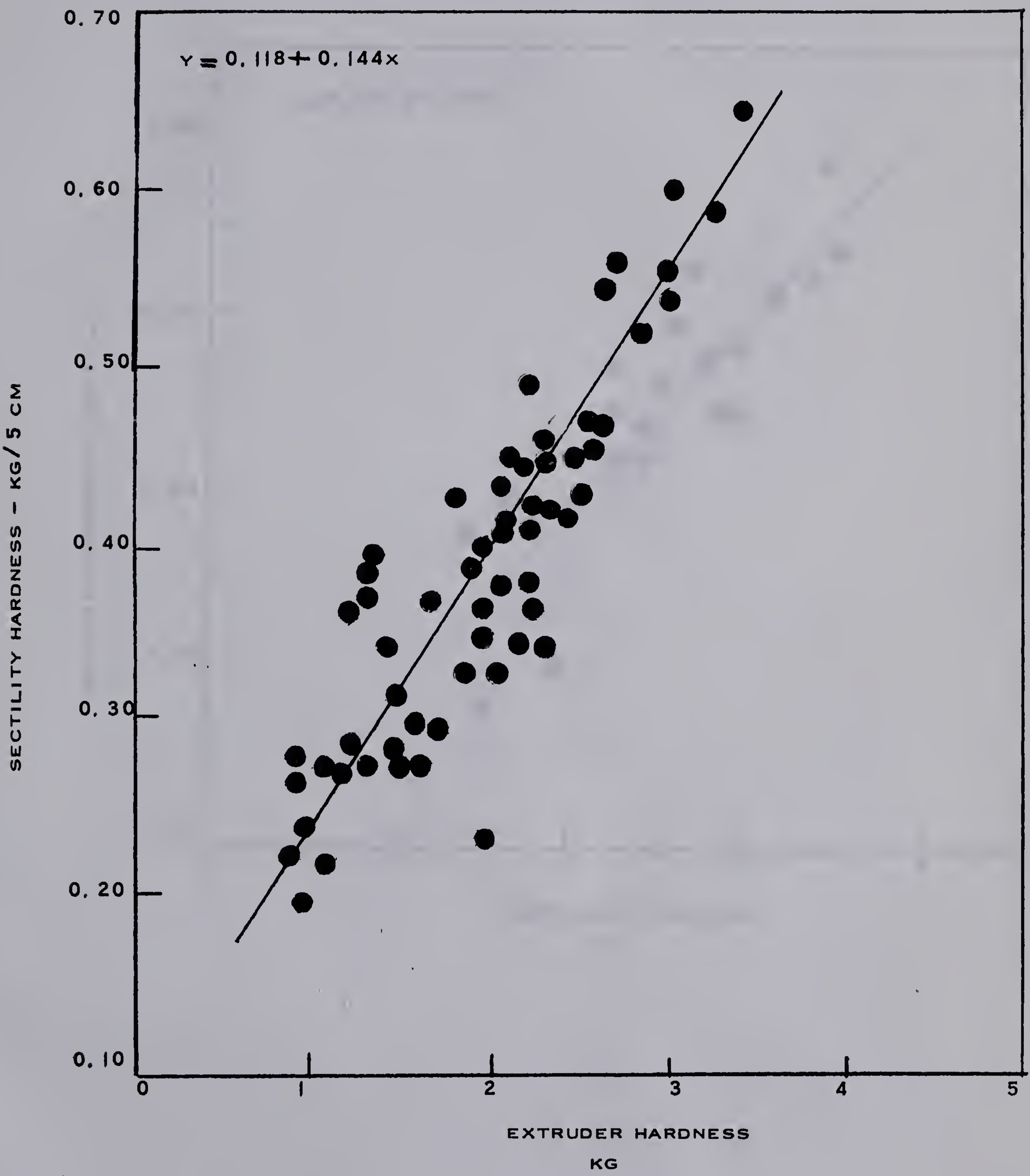


FIGURE A-5 RELATIONSHIP BETWEEN EXTRUSION AND SECTILITY VALUES FOR 20 SAMPLES OF CONTINUOUS BUTTER

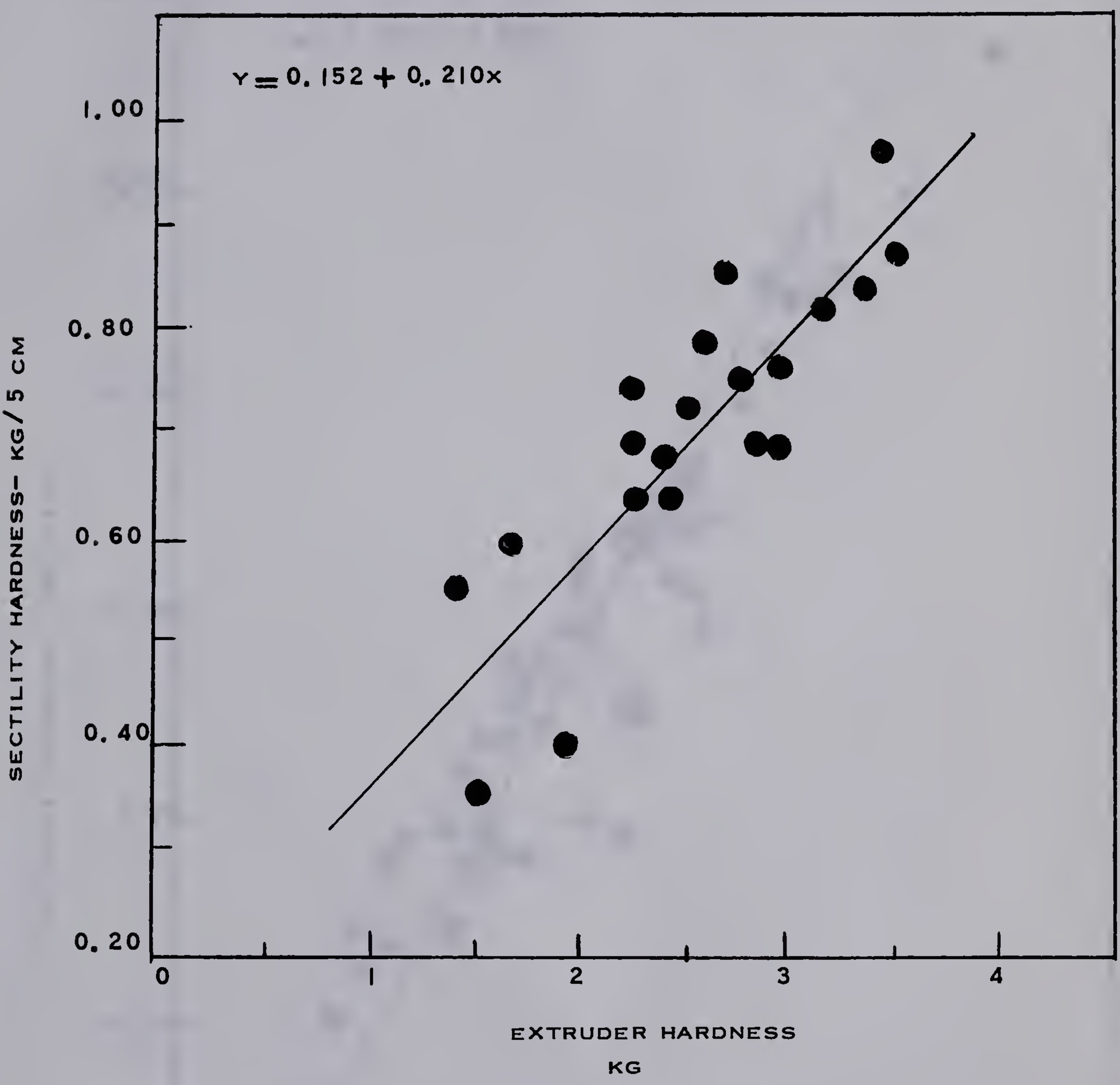


FIGURE A-6 RELATIONSHIP BETWEEN PENETRATION AND SECTILITY VALUES FOR 60 SAMPLES OF CONVENTIONAL BUTTER

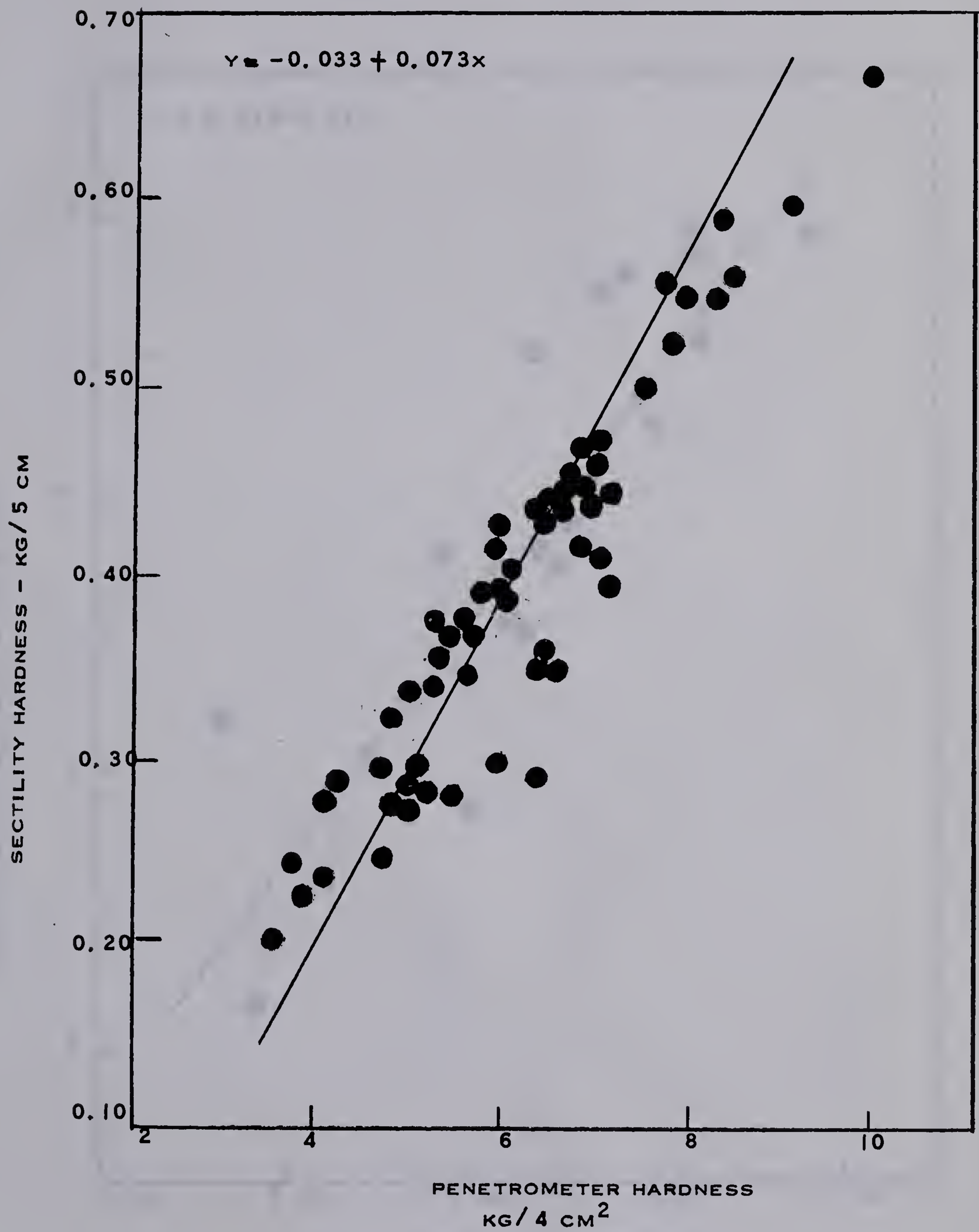


FIGURE A-7 RELATIONSHIP BETWEEN PENETRATION AND SECTILITY VALUES FOR 20 SAMPLES OF CONTINUOUS BUTTER

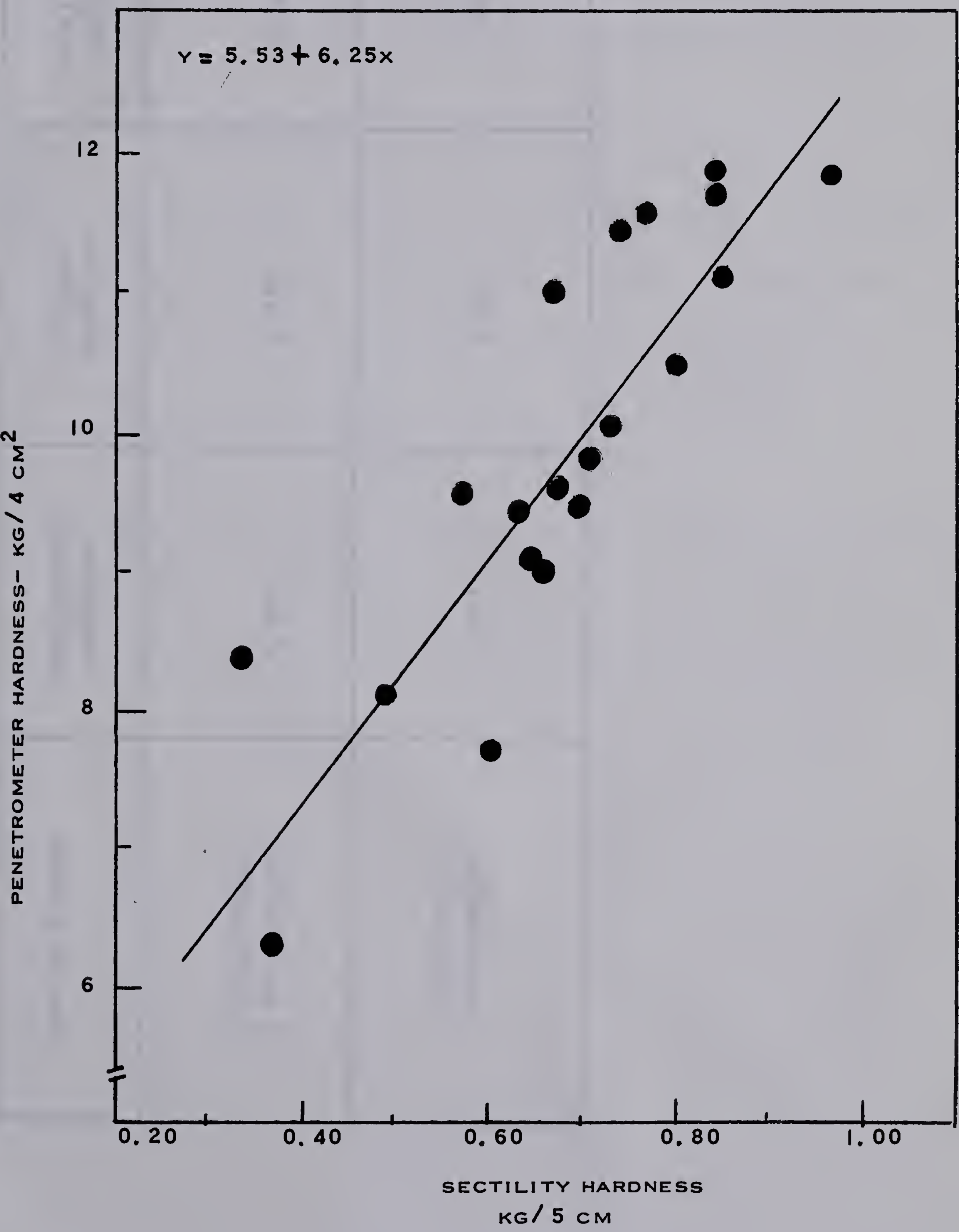


TABLE A-2 CORRELATION COEFFICIENTS SIGNIFICANT AT 1% LEVEL FOR CONVENTIONAL AND CONTINUOUS BUTTERS TESTED FOR HARDNESS BY THREE METHODS

TYPE OF BUTTER	PENETRATION VS EXTRUSION	EXTRUSION VS SECTILITY	PENETRATION VS SECTILITY
CONVENTIONAL [60 SAMPLES]	0.905	0.856	0.920
CONTINUOUS [20 SAMPLES]	0.791	0.820	0.638

3. Discussion

The correlations for the three methods of measuring butter hardness were high for the sixty conventional butters tested. The lowest value of r was 0.856 for extrusion vs. sectility; however, this value was well above the coefficient r = 0.325 required for significance at the 1% level for 58 degrees of freedom.

Although a limited number of continuous butters were tested, reasonably high correlations were found for two relationships. The third, penetration vs. sectility, was low (0.638), probably because deviations from linearity occurred when the penetration values were greater than 10 kg/4 cm^2. However, all r values were above r = 0.561 required for significance at the 1% level for 18 degrees of freedom.

From the results obtained, it appears that these methods measure the same property of butter. Testing of a larger number of samples would have resulted in higher coefficients of correlation for continuous butter.